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Evaluation of the Resistance of a Chlorinated Polyethylene Protective Garment Material to Permeation and Degradation by Liquid Chemicals

JRB Associates, Inc., McLean, VA

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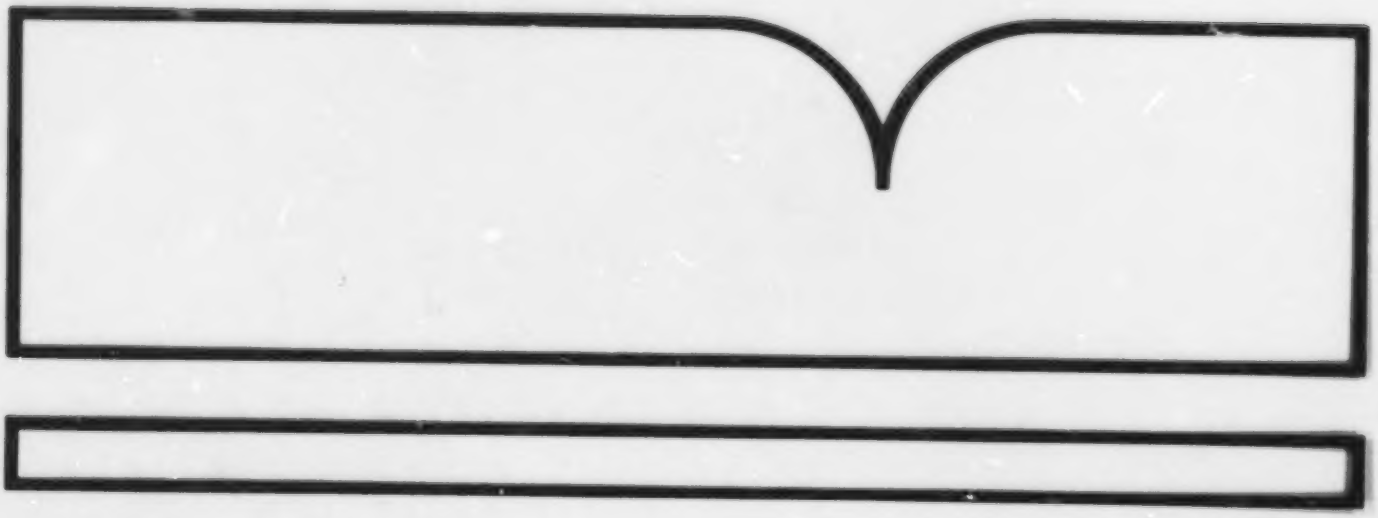

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EVALUATION OF THE RESISTANCE OF A CHLORINATED POLYETHYLENE PROTECTIVE GARMENT MATERIAL TO PERMEATION AND DEGRADATION BY LIQUID CHEMICALS

by

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16. ABSTRACT

The objectives of this project were to investigate existing permeation and degradation resistance data for chlorinated polyethylene (CPE); to develop a laboratory test plan consistent with American Society for Testing and Materials (ASTM) Standard Test Methods F739-81 and D471-79 regarding permeation, solubility, swelling, and degradation of CPE fabric by test chemicals; and to obtain laboratory test data for chemical and CPE combinations of interest for which only limited or questionable permeation and degradation resistance data exist. Ten liquid chemicals (acetic acid, acetic anhydride, acetone, bis(2-chloroethyl)ether, carbon tetrachloride, ethylene diamine, isopropyl alcohol, N-nitrosodimethylamine, phenol, and o-xylene) were tested to determine permeation rates, breakthrough times, and swelling when contacted with 20 mil thick CPE. Mean permeation breakthrough times ranged from 15 minutes to over 8 hours. Steady-state permeation rates ranged from 0 to 170 $mg \cdot m^{-2} \cdot min^{-1}$. Solubility and swelling data also showed wide variation depending upon the chemical. Soaking the CPE in one chemical resulted in a loss of weight, while contact with four other compounds resulted in weight gains by the CPE test swatch of over 100%. Chemical contact was also shown in 8 of 10 cases to reduce the CPE's capacity for resisting tearing due to deformation.

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This report is intended to present and analyze data from protective garment material tests conducted by Rocky Mountain Analytical Laboratory and Radian Corporation. It is not intended to recommend either for or against the use of 20 mil chlorinated polyethylene as an outergarment material for protection from harmful exposures. Such recommendations should only be made by qualified personnel based upon consideration of the data provided in this report and other pertinent data and information about the exposure situation.

FOREWORD

Today's rapidly developing and changing technologies and industrial products and practices frequently carry with them the increased generation of solid and hazardous wastes. These materials, if improperly dealt with, can threaten both public health and the environment. Abandoned waste sites and accidental releases of toxic and hazardous substances to the environment also have important environmental and public health implications. The Hazardous Waste Engineering Research Laboratory assists in providing an authoritative and defensible engineering basis for assessing and solving these problems. Its products support the policies, programs and regulations of the Environmental Protection Agency, the permitting and other responsibilities of State and local governments and the needs of both large and small businesses in handling their wastes responsibly and economically.

This report describes a study concerning the chemical permeation and degradation of a 20 mil thick chlorinated polyethylene outer garment material. This report provides useful information and data to (a) health and safety advisors for field investigators who must choose suit fabrics for partial or totally encapsulating suits for use near spilled chemicals or hazardous wastes, and (b) researchers who are evaluating the effectiveness of protective clothing materials for resisting permeation and degradation following chemical contact. For further information, please contact the Land Pollution Control Division of the Hazardous Waste Engineering Research Laboratory.

David G. Stephan, Director
Hazardous Waste Engineering Research Laboratory

ABSTRACT

The objectives of this project were to investigate existing permeation and degradation resistance data for chlorinated polyethylene (CPE); to develop a laboratory test plan consistent with American Society for Testing and Materials (ASTM) Standard Test Methods F739-81 and D471-79 regarding permeation, solubility, swelling, and degradation of CPE fabric by test chemicals; and to obtain laboratory test data for chemical and CPE combinations of interest for which only limited or questionable permeation and degradation resistance data exist. Ten liquid chemicals (acetic acid, acetic anhydride, acetone, bis(2-chloroethyl)ether, carbon tetrachloride, ethylene diamine, isopropyl alcohol, N-nitrosodimethylamine, phenol, and o-xylene) were tested to determine permeation rates, breakthrough times, and swelling when contacted with 20 mil thick CPE. Mean permeation breakthrough times ranged from 15 minutes to over 8 hours. Steady-state permeation rates ranged from 0 to 170 $mg \cdot m^{-2} \cdot min^{-1}$. Solubility and swelling data also showed wide variation depending upon the chemical. Soaking the CPE in one chemical resulted in a loss of weight, while contact with four other compounds resulted in weight gains by the CPE test swatch of over 100%. Chemical contact was also shown in 8 of 10 cases to reduce the CPE's capacity for resisting tearing due to deformation.

This report was submitted in fulfillment of Contract No. 68-03-3113, Task No. 1-1 by JRB/Science Applications International Corporation under the sponsorship of the U.S. Environmental Protection Agency. Permeation, solubility, swelling and degradation testing was conducted by Rocky Mountain Analytical Laboratory under EPA Contract Nos. 68-01-6812, 68-01-6842, 68-01-6876, 68-01-6879. Permeation tests were also conducted by Radian Corporation. An external QA/QC review was performed by IT Corporation under EPA Contract No. 68-03-3069.

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ABBREVIATIONS AND SYMBOLS

A - surface area of the sample material swatch contacted

A_L - surface area of a LSCPE

A_{IR} - absorbance = - log of fractional transmittance

ASTM - American Society for Testing and Materials

C - concentration of the chemical in the collecting medium

C_{BT} - chemical concentration in the collecting medium when the chemical is first detected (e.g., equivalent to the detection limit if continuous monitoring is performed)

CCl_4 - carbon tetrachloride

C_{DL} - detection limit

CERCLA - Comprehensive Environmental Response, Compensation, and Liability Act of 1980

cm - centimeters

CPE - chlorinated polyethylene

e - slope of the calibration curve divided by the pathlength

EPA - Environmental Protection Agency

g - grams

h - height

IR - infrared

l - spectral cell pathlength

LSCPE - Long-term, Self-contained, Chemical Protective Ensemble which is being developed under EPA/Army Interagency Agreement DW 21930850

m - meters

mg - milligrams

mm - millimeters

OHMTADS - Oil and Hazardous Materials Technical Assistance Data System

OSHA - Occupational Safety and Health Administration

r - radius

RMAL - Rocky Mountain Analytical Laboratory

R_S - skin absorption rate

S - slope of the linear section of the breakthrough curve

sec - second

t - time

TLV(R) - threshold limit value

TWA - time weighted average

um - micrometers

ACKNOWLEDGEMENTS

The performance of permeation, solubility, swelling, and degradation tests under the direction of Chuck Wibby of Rocky Mountain Analytical Laboratory is gratefully acknowledged. The performance of an external audit by Robyn Wagner and Janet Cullinane of IT Corporation, Knoxville, Tennessee, is also much appreciated. As part of the external audit, duplicate permeation tests were performed by Radian Corporation, Austin, Texas. Thanks are also extended to ILC Dover, Frederica, Delaware for providing 20 mil thick chlorinated polyethylene test samples.

SECTION 1

INTRODUCTION

PROJECT OBJECTIVES/APPROACH/ORGANIZATION

The objectives of this project were to evaluate the permeation and degradation resistance of a 20 mil chlorinated polyethylene (CPE) chemical protective outer garment material when contacted by hazardous and toxic substances to which chemical spill and uncontrolled hazardous waste site response personnel may be exposed. To reach this objective, JRB Associates identified and evaluated the existing permeation and degradation data base as of August 1983; proposed a prioritized list of untested substances; and developed a preliminary laboratory permeation/degradation resistance test plan, which was based primarily on ASTM Method D-471-79, "Standard Test Method for Rubber Property - Effects of Liquids" and ASTM F-739-81, "Standard Test Method for Resistance of Protective Clothing Materials to Permeation by Hazardous Liquid Chemicals"; and managed the execution of this test plan by Rocky Mountain Analytical Laboratories (RMAL). An external audit of the permeation testing was conducted by IT Corporation-EERU. As part of the external audit, duplicate samples were subjected to permeation testing by Radian Corporation.

Rationale for Evaluating the Performance of 20 mil CPE

The U.S. Environmental Protection Agency (EPA), pursuant to the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) participates in response, investigation, mitigation, control, and cleanup of hazardous substance spills and uncontrolled hazardous waste sites. When conditions require it, EPA personnel utilize totally encapsulating ensembles to provide protection from dermal and respiratory contact with hazardous substances. Based on their field experiences with totally encapsulating ensembles, EPA personnel expressed a need in 1981 for an ensemble with a self-contained, long term air supply; breathing air and body cooling; and total body protection from chemical splashes or vapors and from pinholes or leaking seals in the protective outer garment. The U.S. Army's Chemical Systems Laboratory (since re-named the Chemical Research and Development Center) and the U.S. Coast Guard had developed a prototype ensemble that potentially met EPA's requirements (Sandy, 1981). The EPA entered into an Interagency Agreement with the U.S. Army in 1982 to advance the development of the prototype ensemble, which is named the Long-term, Self-contained, Chemical Protective Ensemble (LSCPE) (Royer, et al., 1984).

The current LSCPE outer garment consists primarily of 20 mil chlorinated polyethylene (CPE), which had been previously selected by the Army after

extensive evaluation and testing for use in its Demilitarization Protective Ensemble (English, 1982). However, the outer garment only required that penetration testing be conducted with "(chemical warfare) agents, decontaminants, hydraulic fluids, and other liquid substances present in the toxic area and liable to come into contact with the ensemble."[1] It was clear that for EPA's intended use of the LSCPE, additional data were required on the degradation and permeation resistance of CPE when in contact with chemical substances that EPA personnel were likely to encounter at chemical spills and uncontrolled hazardous waste sites.

The U.S. Coast Guard had conducted a study (Friel, et al., 1980) which indicated that butyl rubber, a widely-used outer garment material, would not provide adequate degradation and/or permeation resistance against approximately 200 of 400 chemicals in commerce for which it was determined that totally encapsulating outer garments would be required for effective spill response. The Coast Guard initiated a second study (Algera, 1982) to identify outer garment materials that would provide adequate degradation and permeation resistance for the substances against which butyl rubber failed, and CPE was one of the outer garment materials that was evaluated in that study. It was found that CPE would provide satisfactory protection against some, but not all, of the chemical substances for which butyl rubber was not acceptable.

Although CPE had known incompatibilities, it was also clear that (a) no existing outer garment material was suitable for all chemical substances, (b) the Army had conducted a wide-ranging evaluation of other outer garment materials before selecting CPE for its chemical agent protective outer garment, and (c) the relatively low cost of CPE was a significant advantage since the difficulty of decontaminating outer garments can make it necessary to dispose of the outer garment before it is physically worn out. Furthermore, the U.S. Coast Guard has continued to evaluate additional outer garment materials and coordination between EPA and the Coast Guard has been maintained with the intent of ensuring that breathing and cooling apparatus of the LSCPE are compatible with new outer garments developed by the Coast Guard program.

Overview of Work Described in this Report

An investigation, which is the subject of this report, was initiated by EPA, under Contract 68-03-3113 with JRB Associates, of the resistance of the primary LSCPE outer garment material (20 mil CPE) to degradation, solubility, swelling, and permeation, when contacted by liquid chemicals. The substances that were evaluated were substances (a) that were likely to be encountered at chemical spills or uncontrolled hazardous waste sites and (b) for which test data were not available. JRB Associates identified and evaluated the existing permeation/degradation resistance data base; proposed a prioritized list of untested substances for EPA approval; developed a preliminary laboratory permeation/degradation resistance test plan; and managed the execution of this plan by Rocky Mountain Analytical Laboratories (RMAL). Permeation tests were also conducted by Radian Corporation. An

[1] The Demilitarization Protective Ensemble, Volume I - The Development Program, An Overview. U.S. Army Chemical Systems Laboratory, 1982, p. 24.

external QA/QC audit of the permeation tests was conducted by IT Corporation-EERU.

The primary target audience for the data provided in this report consists of EPA personnel involved in the selection and use of the LSCPE outer garment or other outer garments constructed of similar 20 mil chlorinated polyethylene (CPE). This report is also expected to be of interest to U.S. Coast Guard and U.S. Army chemical protective clothing programs and to organizations that are assembling data bases on chemical protective clothing performance.

This report specifically describes the results of solubility and swelling, degradation, and permeation tests that were conducted with 20 mil thick CPE against 10 liquid chemicals (acetic acid, acetic anhydride, acetone, bis(2-chloro-ethyl)ether, carbon tetrachloride, ethylenediamine, isopropyl alcohol, N-nitrosodimethyl amine, phenol, and xylene. The data reported include the length, width, thickness, and mass changes that occurred in CPE test swatches that were immersed in the test liquids. Also reported is the comparative resistance of chemical-soaked versus clean samples to failure via tearing when subjected to repeated prodding with a blunt rod. The permeation resistance of 20 mil CPE is reported as breakthrough times (i.e. the time at which a detectable quantity of contaminant first appeared on the unexposed side of the CPE swatch) and steady-state permeation rates (i.e., the steady-state flux ($mg \cdot m^{-2} \cdot min^{-1}$) of hazardous liquid across the membrane). Tests were conducted only on 20 mil CPE, which is the principal component of the LSCPE outer garment. Tests were not conducted on other areas of the LSCPE outer garment, such as visors, vents, seams, or areas covered by materials other than CPE.

SECTION 2

CONCLUSIONS

The Long-term, Self-contained, Chemical Protective Ensemble (LSCPE), which has an outer garment that is constructed primarily of 20 mil CPE, has a self contained breathing air supply of 2.5 hours (150 minutes). Thus, one criterion for fully satisfactory performance of the CPE is that it be capable of resisting permeation by the test chemical for 150 minutes or more. The second criterion for judging the performance of CPE was that chemical contact should not seriously degrade the tearing resistance of the CPE after repeated prodding with a blunt steel rod.

- CPE (20 mil thick) demonstrated high resistance to both permeation and tearing when contacted by carbon tetrachloride and isopropanol.

  Four of the compounds that were tested, [isopropanol (> 480 min*), carbon tetrachloride (207 min*), phenol (175 min), and ethylenediamine (160 min)] had mean breakthrough times in excess of 150 minutes, and acetic acid (144 min) nearly met the 150 minute criterion. However, of the five compounds cited immediately above, only isopropanol and carbon tetrachloride did not degrade the ability of CPE to resist tearing after being subjected to repeated deformation. Phenol exposure caused serious loss of tearing resistance -- each of three CPE samples stiffened and tore after repeated deformation. A less severe reduction in tearing resistance was caused following exposure to ethylenediamine and acetic acid.

- One compound, acetone, had a mean breakthrough time (15 min) that would pose problems for an LSCPE wearer, who would probably have to exit a contaminated work area and undergo decontamination prior to doffing the outer garment.

- The mean permeation breakthrough times (42 to 87 minutes) for the remaining compounds [N-nitrosodimethylamine, o-xylene, acetic anhydride, and bis (2-chloroethyl) ether] indicate that moderate protection from permeation is provided. However, only acetic anhydride did not degrade the tearing resistance of CPE. N-nitrosodimethylamine, o-xylene, and bis (2-chloroethyl) ether caused each CPE sample (three samples per chemical) to tear when subjected to repeated prodding with a blunt steel rod.

---

* Breakthrough times of <1 minute were also noted for isopropanol and carbon tetrachloride. Based on data for similar compounds and for the same compounds tested by different laboratories, it is thought likely that these very low breakthrough times are probably due to material imperfections or experimental error, although this has not been conclusively demonstrated.

SECTION 3

MATERIALS AND EXPERIMENTAL PROCEDURES

This section describes the equipment, chemicals, and procedures used in testing the CPE material. Subsection 3.1 contains a discussion on the physical characteristics of the CPE test samples and of the ten challenge chemicals. It also contains a brief discussion on the criteria used in selecting chemicals for testing. Subsections 3.2, 3.3 and 3.4 contain discussions on the apparatus and experimental procedures used to conduct tests for breakthrough and permeation, solubility and swelling, and degradation.

3.1 CHARACTERIZATION OF THE CHLORINATED POLYETHYLENE TEST SAMPLES AND OF THE TEN CHALLENGE CHEMICALS

Test samples of CPE were provided by ILC Dover. The sample material was designated as 20 mil (.020 in) Cloropel™ alloyed CPE (ILC part number ST9C019-03, lot 2). All swatches were smooth, without seams. ILC Dover provided information on the weight and thickness of the CPE and on the types of physical tests to which the material is normally subjected:

WEIGHT	16-20 oz/yd^2
THICKNESS	0.018-0.022 inches (18-20 mil)
TENSILE (lb/in^2)	MACHINE 1950 minimum TRAVERSE 1750 minimum
ELONGATION (90)	MACHINE 275 minimum TRAVERSE 375 minimum
TEAR (lb/in^2)	MACHINE 300 minimum TRAVERSE 275 minimum
SEAM STRENGTH (lb/in-peel)	16 minimum

The selection of the challenge chemicals to be tested was based primarily upon a literature search. A list of 50 chemicals commonly found in hazardous chemical spills and at uncontrolled hazardous waste disposal sites are shown in Table 1. Literature reviewed for this project is listed in the References and Bibliography sections.

TABLE 1. HAZARDOUS CHEMICAL LISTS USED FOR CHOOSING TEST CHEMICALS

	Top 20 Organics Spilled	Top 50 Organics Spilled	Found Most Often at Superfund Sites JRB[2]	Found Most Often at Superfund Sites EERU[3]	OHMTADS[4] List	In Top 50 of Ranked Coast Guard List[5]	Chemical Class
1. Aroclor 1254 (PCB's)	X		X	X			Chlorinated aromatic
2. Isopropyl alcohol	X		X	X			Alcohol
3. Methanol	X		X	X			Alcohol
4. Xylene	X		X	X			Aromatic hydrocarbon
5. Styrene	X		X	X			Aromatic hydrocarbon
6. Acetic acid	X					X	Carboxylic acid
7. Phenol	X						Phenol
8. Acetone	X						Ketone
9. Toluene Diisocyanate	X					X	Isocyanate
10. Methacrylic acid	X						Carboxylic acid
11. Methyl methacrylate	X						Ester
12. Acetic anhydride	X						Carboxylic anhydride
13. Vinyl chloride			X		X	X	Chlorinated olefin
14. bis(2-Chloro-ethyl) ether			X	X			alpha-Halo ether
15. Methylene chloride		X	X				Chlorinated aliphatic
16. Chlordane		X					Chlorinated alicyclic
17. Carbon tetra-chloride		X		X		X	Chlorinated aliphatic
18. Nitrobenzene					X		Nitroaromatic
19. Aniline		X	X				Aromatic amine
20. Tricresyl phosphate				X		X	Organophosphate

(continued)

TABLE 1. HAZARDOUS CHEMICAL LISTS USED FOR CHOOSING TEST CHEMICALS (continued)

	Top 20 Organics Spilled	Top 50 Organics Spilled	Found Most Often at Superfund Sites JRB[2]	Found Most Often at Superfund Sites EERU[3]	OHMTADS[4] List	In Top 50 of Ranked Coast Guard List[5]	Chemical Class
21. N-Nitrosodi-methylamine				X			Nitroaromatic
22. Benzyl benzene				X			Aromatic
23. Methyl isobutyl ketone				X			Ketone
24. 2,4-Dichloro-phenol				X			Chlorophenol
25. Lindane			X				Chlorinated alicyclic
26. Fluoranthene			X				Polynuclear aromatic
27. Pentachloro-phenol			X				Chlorophenol
28. Urethane							
29. Formic acid		X					Amide
30. Acetanilide		X				X	Carboxylic acid
31. Pyridine		X					Aromatic amide
32. Malathion		X				X	Heteroaromatic
33. Dimethyl sulfate							Organophosphate
34. Chloropicrin						X	Alkyl sulfate
35. Tetraethyl lead						X	Chloronitro-aliphatic
36. Dichlorvos						X	Organometallic
37. Tetramethyl lead						X	Organophosphate
38. Benzoyl chloride						X	Organometallic
39. Dioxane						X	Acid chloride
40. Allyl chloride			X			X	Ether
41. Ethylene oxide						X	Chlorinated olefin
42. Cumene hydro-peroxide						X	Epoxide
43. Methyl hydrazine						X	Peroxide
44. Parathion						X	Hydrazine
45. Thioglycolic acid						X	Organophosphate
46. Trichloreacetic acid						X	Mercapto-acide

(continued)

TABLE 1. HAZARDOUS CHEMICAL LISTS USED FOR CHOOSING TEST CHEMICALS (continued)

	Top 20 Organics Spilled	Top 50 Organics Spilled	Found Most Often at Superfund Sites JRB[2]	Found Most Often at Superfund Sites EERU[3]	OHMTADS[4] List	In Top 50 of Ranked Coast Guard List[5]	Chemical Class
47. Epichlorohydrin						X	Epoxide/chlorinated aliphatic
48. 1,2-Dibromo-3-chloropropane *							Brominated aliphatic
49. Bis (2-ethylhexyl)* phthalate							Phthalate ester
50. Ethylenediamine*							Aliphatic amine

[1] Larry Krasner, Factory Mutual Research Corporation, personal communication, July 1982.

[2] JRB Associates, 1982; Information Search Report for the Chemical Countermeasures Program; for EPA/MERL/OHMSB

[3] Emergency Environmental Response Unit, Rodney Turpin from ERT Sorbent Study

[4] Oil and Hazardous Materials Technical Assistance Data System

[5] ILC Dover, 1982; Development of a Hazardous Chemical Protective Ensemble; for U.S. Coast Guard

* Selected as functional group representatives.

This report documents the testing of ten of the Table 1 chemicals that were subjected to breakthrough and permeation, solubility and swelling, and degradation tests. These ten chemicals are listed in Table 2.

3.2 METHODOLOGY FOR DETERMINING BREAKTHROUGH TIMES AND PERMEATION RATES OF TEST CHEMICALS THROUGH CHLORINATED POLYETHYLENE

Breakthrough times and permeation rate measurements by RMAL and Radian Corporation were performed, with minor variations, according to ASTM "Standard Test Method for Resistance of Protective Clothing Materials to Permeation by Hazardous Liquid Chemicals" (F739-81), which is in Appendix A.

3.2.1 RMAL Methodology

3.2.1.1 Testing was specifically performed as described in Appendix C. A one-inch (diameter) permeation test cell was used for all testing instead of the two-inch cell called for in (ASTM F 739-81). The one-inch cell (Pesce Lab Sales, Kennett Square, PA) was substituted to minimize the amount of challenge chemical required for each test. Although method detection limits were generally higher than what could have been attained with the two-inch cell, the substitution was viewed as appropriate in light of the extremely hazardous nature of some of the challenge chemicals.

3.2.2 Radian Methodology

3.2.2.1 All permeation data provided by Radian Corporation was generated according to ASTM F 739-81. A one inch test cell was substituted for the two inch test cell specified in the method.

The testing of CPE against acetic acid, acetic anhydride, and ethylene diamine was performed using water as the collecting medium. Deionized water was circulated continuously through the collection cell to a conductivity detector for the determination of breakthrough time and steady state rate. The testing of phenol against CPE also employed water as the collecting medium. Discrete samples were removed at regular time intervals for analysis by flame ionization-gas chromatography, (FID-GC). The remaining six challenge chemicals were tested using a nitrogen gas collecting system that was directed to an FID-GC system for continuous monitoring.

The detection limits provided for each chemical represent the lowest concentration of a challenge chemical present in the collecting medium. This value is determined from a plot of concentration vs response using zero and the low concentration standard. The detection limit is equivalent to any instrumental response greater than ten percent of background noise.

3.3 METHODOLOGY FOR MEASURING THE SOLUBILITY AND SWELLING OF CPE CAUSED BY IMMERSION IN LIQUID TEST CHEMICALS

3.3.1 Introduction

ASTM Method D471-79 with minor variations was used by RMAL to determine the solubility and swelling of the CPE material after immersion into the

ten challenge chemicals listed in Table 2. The total immersion option in the method was chosen (not the single-sided immersion procedure).

3.3.2 Apparatus

Thickness gage, B.C. Ames #2 Comparator, B.C. Ames Co., Waltham, MA
Balance, Mettler PE 160, Mettler Instrument Corp., Hightstown, NJ
Test Tubes, 38 mm od, Fisher Scientific Co., Pittsburgh, PA.
Vernier calipers, readable to 0.001 inch, American Scientific Products, catalog #M1110, Denver CO.

3.3.3 Procedure

1. Cut the CPE material into 1" x 2" rectangles (25 mm x 50 mm).

2. Determine the thickness of each swatch in triplicate using the procedure outlined in Federal Standard Number 191A (Determination of Thickness of Materials) and record.

3. Determine the width and length of each swatch in triplicate at three different points using the vernier calipers. Record to the nearest 0.001 inch.

4. Determine the mass of each swatch using the Mettler PE 160 balance and record.

5. Fill the test tube to a depth that will permit the swatch to be completely immersed and record the temperature of the challenge chemical.

6. Immerse the CPE swatch in the challenge chemical and stopper the test tube. At specific intervals (5 min., 15 min., 30 min., 60 min., 3 hours, 24 hours), remove each swatch from the challenge chemical, blot dry, and repeat steps 2-4.

7. Note any changes in the physical appearance of the swatch.

8. Continue to test until the swatch is saturated. Saturation is indicated by successive mass measurements differing by less than 2%. This criterion does not apply if the mass has not changed from the initial value recorded in step 4. If saturation of the swatch is not complete in 24 hours, terminate the testing.

9. Terminate the test if the swatch deteriorates to the point that meaningful determinations cannot be made.

10. Complete steps # 2 through # 9 three times. Each time, use a new CPE swatch and fresh chemical.

3.3.4 Calculations

1. Average all determinations that were made in triplicate (width, length, thickness).

TABLE 2. TEST COMPOUNDS USED BY RMAL AND RADIAN

TEST COMPOUNDS, RMAL

COMPOUNDS	PURITY (%)
Acetic acid	99.9
Acetic anhydride	100.2
Acetone	NA
Bis(2-choloroethyl) ether	NA
Carbon tetrachloride	NA
Ethylenediamine	98.8
Isopropyl alcohol	NA
N-Nitrosodimethylamine	99+
Phenol	91%, oxalic acid preservative
o-Xylene	NA

NA = Not available.

TEST COMPOUNDS, RADIAN

COMPOUNDS	PURITY (%)
Acetic acid	99.8
Acetic anhydride	99+
Acetone	99
Bis(2-choloroethyl) ether	99
Carbon tetrachloride	99
Ethylenediamine	99
Isopropyl alcohol	99
N-Nitrosodimethylamine	99
Phenol	85
o-Xylene	97

NA = Not available.

2. Calculate the change in mass using the following formula:
   change in mass, % = $(M_2 - M_1)/M_1 \times 100$
   where: M_1 = initial mass of specimen, g
   M_2 = mass of specimen, g, after immersion

3. Calculate the change in dimensions as follows:
   change in length, % = $(L - L_0)/L_0 \times 100$
   where: L_0 = average initial length of specimen
   L = average length after immersion
   Calculate the change in width and the change in thickness in a similar manner.
   Sample Calculation:

Aniline

Change in mass, % = (1.2 g - 0.87 g)/0.87 g X 100%
= 43%

Change in length = (6.05 cm - 5.28 cm)/5.28 cm X 100%
= 14%

3.4 METHODOLOGY FOR MEASURING THE DEGRADATION OF CPE FOLLOWING IMMERSION IN LIQUID CHEMICALS

This test was designed and conducted by RMAL to measure the degradation of chemical-soaked CPE when subjected to repeated physical deformation. CPE swatches were immersed into the liquid test chemicals using the same procedures as those described in Section 3.3. Every swatch was then subjected to prodding to a depth of 0.5 inches by a blunt steel rod at a rate of 10 $\pm$ 2 times per minute. Every hour, for up to two hours, the swatch was inspected and observed for changes in color, surface smoothness or shape with respect to a control swatch that had not been saturated with the test chemical, but had been subjected to the same repeated prodding.

The apparatus used to subject the CPE swatches to deformation consisted of an electrically driven motor connected to a series of belts and pulleys. The pulleys were chosen to produce the desired rate of prodding, and to allow the blunt rod to prod the swatch to a depth of 0.5 inches (13 mm). After a swatch was saturated with the test chemical, it was held perpendicular to the prodding rod with a 1" brass union.

SECTION 4

RESULTS AND DISCUSSION

Subsections 4.1 through 4.4 contain the results and corresponding discussion of the breakthrough time and permeation rate tests, the solubility and swelling tests, and the degradation tests, respectively. Subsection 4.5 relates the test performance of CPE to potential operational requirements of the LSCPE wearers.

4.1 BREAKTHROUGH TIMES AND PERMEATION RATES OF TEST CHEMICALS THROUGH CPE

Breakthrough times and steady-state permeation rates for the ten test chemicals passing through the CPE test swatches are listed in Tables 3 and 4. Mean breakthrough times from both laboratories are in relatively close agreement between 8 of the 10 pairs of data. There was a major difference between the mean breakthrough times for acetic acid (237 min vs. 144 min). No explanation for the discrepancy was found, but both mean breakthrough times represent significant resistance to permeation. There was also a major difference (<1 min vs. >480 min) between the individual laboratory mean breakthrough times for isopropanol. The low (<1 min) values were discarded in the evaluation of CPE's performance since (a) CPE's resistance to another alcohol (methanol) was found from other data to be much greater than one minute, (b) CPE did not show signs of degradation as a result of contact with isopropanol, and (c) similar very short breakthrough times for carbon tetrachloride were generated and were discarded since good precision, both within and between laboratories, was demonstrated for high breakthrough times that were generated for the carbon tetrachloride. It is assumed that the cause of the low breakthrough times for isopropanol are the same as for the carbon tetrachloride.

In the case of carbon tetrachloride, breakthrough times from one lab fell into two distinct groups, <1 minute and 183-245 minutes. The second laboratory's data were in the 205-210 minute range. The low breakthrough times were discarded due to the close agreement on the high values between two independent laboratories.

4.2 EXTERNAL QUALITY ASSURANCE PROGRAM FOR PERMEATION TESTS

4.2.1 Introduction

The role of IT Corporation-EERU was to conduct an external quality assurance audit of the permeation testing. The purpose of the program was to ensure an acceptable degree of confidence in the data generated during

TABLE 3. BREAKTHROUGH TIMES OF TEST CHEMICALS THROUGH 20 mil CPE

Chemical	RMAL Breakthrough Time (min) Raw	Mean	R.S.D. %	Radian Breakthrough Time (min) Raw	Mean	R.S.D. %	RMAL and Radian Mean Breakthrough Time (min)	R.S.D. %
Acetic Acid	265 241 205	237	12.7	140 146 147	144	2.6	191	28.5
Acetic Anhydride	86 76 64	75	14.6	72 72 72	72	0	74	9.8
Acetone	16 18 18	17	6.7	15 15 15	15	0	16	9.1
Bis-(2 Chloroethyl)ether	99 56 61	72	32.7	91 83 87	87	4.6	80	21.6
Carbon Tetrachloride	245 199 183	209	15.4	205 210 207	207	1.2	208	9.8
Ethylene diamine	128 131 101	120	13.3	154 159 167	160	4.1	140	17.3
Isopropanol	N/A	<1	N/A	>480 >480 >480	>480	N/A	N/A	N/A
N-Nitrosodimethylamine	38 32 21	30	28.4	42 42 42	42	0	36	23.2
Phenol	184 224	204	13.5	165 180 180	175	5.0	187	11.8
O-Xylene	77 72 67	72	6.9	62 66 60	63	4.9	67	9.3

TABLE 4. PERMEATION RATES OF TEST CHEMICALS THROUGH 20 mil CPE

Chemical	Permeation Rate $mg \cdot m^{-2}$ Raw	Radian Permeation Rate sec^{-1} Mean	R.S.D.
Acetic Acid	10 10 2	7	65.7
Acetic Anhydride	10 7 9	9	16.7
Acetone	169 146 194	170	14.1
Bis-(2-Chloroethyl) ether	154 45 40	80	80.0
Carbon Tetrachloride	15 14 10	13	20.4
Ethylene diamine	2 7 10	6	63.8
Isopropanol	0 0 0	0	N/A
N-Nitrosodimethylamine	55 84 81	73	21.7
Phenol	9 11 10	10	10.0
O-Xylene	32 29 31	31	5.0

the permeation testing of the 20 mil CPE outer garment material. This was accomplished through the use of performance evaluation samples, interlaboratory testing, and review of analytical protocols, internal quality control procedures, data, and analysis results. A quality assurance systems audit, i.e., an on-site review of RMAL's facilities, equipment, personnel, and procedures, was performed by JRB Associates.

4.2.2 Analysis of Performance Evaluations Samples

In order to obtain some measure of the acceptability of the permeation testing as performed by RMAL, it was requested that they test via ASTM F-739-81, the performance of a neoprene swatch vs. acetone. This sample was from the lot previously used by ASTM during the round-robin testing of ASTM F739-81. The round-robin validation of this method involved the testing of a neoprene test swatch against acetone using water as the collecting medium and analysis of discrete samples by flame ionization-gas chromatography. A summary of the round-robin results is included in Table 5. RMAL substituted an infra-red monitoring system and a gas collecting medium for the water collection and FID-GC analysis used during the interlaboratory study. They also used a one inch test cell instead of the two inch cell. Results submitted by RMAL for breakthrough time were within the range of the results obtained during the round-robin study; however, the initial results submitted by RMAL for steady state permeation rate were unacceptable. After a review of their calculations, RMAL submitted amended results which were well within the range of the other laboratories. IT-EERU also performed permeation testing of the neoprene vs. acetone as an additional quality assurance measure. A summary of the results submitted by RMAL and IT-EERU are also presented in Table 5.

TABLE 5. PERFORMANCE EVALUATION SAMPLE RESULTS

Laboratory	Breakthrough Time (min)	Permeation Rate $mg \cdot m^{-2} \cdot min^{-1}$
Round-Robin Laboratories	10-20 ($\bar{x}$ = 13)	933-3301 ($\bar{x}$ = 2292)
Rocky Mountain Analytical Lab.	11	2200
IT Corporation-EERU	14	2868

4.2.3 Split Sample Analysis

Another aspect of the external quality assurance program involved an interlaboratory comparison of the permeation resistance of CPE against ten chemicals. The ten chemicals were selected for the wide variety of chemical classes they represent and because several showed "double breakthrough

times"[1] during the testing performed by RMAL. They included acetic acid, acetic anhydride, acetone, bis-chloroethylether, carbon tetrachloride, ethylenediamine, isopropyl alcohol, N-nitrosodimethylamine, phenol, and o-xylene. Radian Corporation performed the duplicate testing using a gas collection system with gas chromatography flame ionization detection.

Tables 3 and 4, which have been shown earlier in this section, list the breakthrough time and steady state rate reported by Radian and recalculated results for RMAL. Precision is reported as relative standard deviation of the triplicate tests performed by each laboratory, as well as for their combined results. Comparison of results between the two laboratories for breakthrough time is dependent upon the detection limits obtained by the two laboratories. This is especially important in this study since different analytical techniques were employed. Radian determined detection limit experimentally, however, RMAL did not. Therefore, approximate detection limits for each chemical were determined as any response (absorbance) greater than the peak height of the instrument noise. Table 6 lists the detection limits obtained by Radian and the detection limits approximated for RMAL.

4.2.4 Data Review

All testing was performed in triplicate and, for the most part, in accordance with ASTM F739-81. One major change in the method was the use by RMAL of a 1 inch test cell instead of the 2 inch cell. However, since the results from the performance evaluation sample were acceptable, it was concluded that the size of the test cell was irrelevant. Also, in calculating the steady state permeation rate, the slope of a line representing concentration vs. time was determined using only two points. Although ASTM F739-81 does not specify a method for determining slope, it is inherently more accurate to use several points and calculate slope by linear regression. Recalculation of the steady state rate using more than two points showed a significant change in some cases.

The calibration data used by RMAL to calculate concentration of challenge chemical in collecting medium were not fully acceptable. The instrument was only calibrated for a narrow concentration range, and samples with responses above and below the range were quantified even though linearity was not proven. In several cases, the linearity of the narrow calibration range is questionable.

Several calculation errors were detected and corrected as a result of the audit of the RMAL data.

Baseline drift was evident during RMAL's testing of several chemicals (e.g., five of seven tests with carbon tetrachloride). In cases where baseline drift occurred it was difficult to decipher drift from actual

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[1] The preliminary reports provided by RMAL showed an immediate breakthrough and slow rate followed by a leveling off of the baseline and then another, sharper breakthrough for carbon tetrachloride, o-xylene and N-nitrosodimethylamine.

TABLE 6. DETECTION LIMITS

Chemical	RMAL		Radian	
	ul/l#	mg/l†	ul/l	mg/l
Acetic Acid	.013	.014	.02	.02*
Acetic Anhydride	.008	.009	.02	.02*
Acetone	.032	.025	.2	.005
Bis-2-Chloroethylether	.011	.013	2	.011
Carbon Tetrachloride	.007	.011	1	.006
Ethylenediamine	.305	.274	1	1.0*
Isopropanol	----	----	.1	.0003
N-Nitrosodimethylamine	.107	.108	.6	.0018
Phenol	.069	.074	53	53.0*
O-Xylene	.147	.128	.05	.0002

= ul gaseous challenge chemical/L nitrogen or He gas

† = mg challenge chemical/L nitrogen or He gas

* = water used as the collecting medium

breakthrough. The drift in baseline also contributed to erroneous results in converting absorbance to concentration. Several chemicals were re-tested by Radian to determine if this drift was actually an initial very slow breakthrough followed by a second sharper breakthrough. Radian did not report an initial breakthrough.

The results for permeation rate and breakthrough time provided by Radian Corporation were generated using good quality control practices. Detection limits were determined experimentally. Their calibration ranges were broad and showed good linearity. They performed spiking experiments to assure that the system was free of leaks and that flow to the detector was adequate, and several points were used to determine steady-state permeation rate.

4.3 SOLUBILITY AND SWELLING OF CHLORINATED POLYETHYLENE CAUSED BY IMMERSION IN THE TEST CHEMICALS

Table 7 lists the percent changes in the length, width, thickness and mass of CPE swatches due to immersion in the test chemicals. If saturation could not be achieved in 24 hours, the tests were terminated. In such cases, the percent changes listed in Table 7 are due to immersion in the test chemical for 24 hours.

Average percent increases in mass after saturation or immersion in the test chemical for 24 hours ranged from 0.5% for ethylenediamine to 126% for bis(2-chlorethyl ether). Test chemicals which caused percent mass increases of less than 100% of the original CPE swatch weight were ethylene diamine (0.5%), isopropyl alcohol (3.5%), acetic acid (29%), phenol (34%) and acetone (57.3%). Test chemicals that caused mass increases exceeding 100% of the original CPE swatch weight were carbon tetrachloride (110%), N-nitrosodimethylamine (112%), o-xylene (112%) and bis(2-chloroethyl) ether (126%).

4.4 PERFORMANCE OF CHEMICAL-SOAKED CPE WHEN SUBJECTED TO REPEATED PHYSICAL DEFORMATION

Table 8 contains observations on the condition of triplicate, chemical-soaked CPE swatches after they had been subjected to repeated physical deformation as described in subsection 3.4. CPE swatches not exposed to any chemical formed shallow indentations, but essentially recovered their initial shape after the test was terminated.

Chemicals which did not cause any of the triplicate trial swatches to tear during the two-hour deformation test period were acetic anhydride, carbon tetrachloride, and isopropyl alcohol. Chemicals which caused only one of the three test swatches to tear during the two hour deformation test were acetic acid, acetone and ethylene diamine. Chemicals which caused more than one of the three trial swatches to tear during the two hour deformation test were bis (2-chloroethyl) ether, N-nitrosodimethylamine, phenol, and o-xylene.

TABLE 7. RESULTS - SOLUBILITY AND SWELLING OF CPE DUE TO IMMERSION IN LIQUID CHEMICALS

CHALLENGE CHEMICAL	LENGTH * OF TEST	LENGTH, PERCENT CHANGE	WIDTH, PERCENT CHANGE	THICKNESS, PERCENT CHANGE	MASS, PERCENT CHANGE
Acetic acid	24 hrs.	-0.4	-7.1	162	27
	24 hrs.	-1.1	-7.0	210	28
	24 hrs.	-1.1	-6.9	173	31
Acetic anhydride	24 hrs.	0.0	-4.2	9.5	6.1
	24 hrs.	-0.8	-1.7	4.8	2.7
	24 hrs.	-0.3	-5.6	4.8	8.2
Acetone	35 min.	1.0	36	57	50
	15 min.	19.2	4.1	57	58
	15 min.	2.4	19	62	64
bis(2-Chloroethyl) ether	50 min.	37	5.7	57	129
	50 min.	27	10	60	125
	50 min.	26	19	52	123
Carbon tetrachloride	110 min.	2.6	22	45	107
	110 min.	-1.8	24	43	116
	110 min.	0.9	21	43	106
Ethylenediamine	24 hrs.	0.5	-3.8	15	-5.0
	24 hrs.	-1.5	-7.5	15	13
	24 hrs.	-3.2	-5.3	15	-6.4
Isopropyl alcohol	24 hrs.	0.6	1.8	0.0	3.1
	24 hrs.	0.0	0.4	0.0	3.7
	24 hrs.	0.8	-0.5	0.0	3.5

(continued)

* If length of test is < 24 hours, this indicates that saturation (i.e., no additional changes in mass) of the CPE swatch occurred. If a test time of 24 hours is listed, then the test was terminated even though significant changes in mass were still occurring between consecutive weighings.

TABLE 7. RESULTS - SOLUBILITY AND SWELLING OF CPE DUE TO IMMERSION IN LIQUID CHEMICALS (continued)

CHALLENGE CHEMICAL	LENGTH * OF TEST	LENGTH, PERCENT CHANGE	WIDTH, PERCENT CHANGE	THICKNESS, PERCENT CHANGE	MASS, PERCENT CHANGE
N-Nitrosodimethylamine	50 min.	2.7	45	61	115
	50 min.	1.4	48	59	112
	20 min.	44	2.7	53	109
Phenol	24 hrs.	-5.9	2.0	19	9.1
	24 hrs.	-9.0	1.0	66	68
	24 hrs.	-4.8	-8.2	50	25
O-Xylene	36 min.	1.9	48	86	116
	36 min.	14	13	71	112
	44 min.	11	29	71	109

* If test length is <24 hours this indicates that saturation (i.e. no additional change in mass) of the CPE swatch occurred. If a test time of 24 hours is listed, the test was terminated even though significant changes in mass were still occurring between consecutive weighings.

TABLE 8. DEGRADATION OF CHEMICAL-SOAKED CPE REPEATEDLY SUBJECTED TO PHYSICAL DEFORMATION

COMPOUND	COMMENTS/OBSERVATIONS
CPE Blank	All three blanks exhibited an indentation within the first hour that grew to a depth of approximately 0.25 inches by the end of the test. No tears or other physical changes were observed.
Acetic acid	Two of the three replicas exhibited a depression approximately 0.4-0.5 inches deep within one hour. The third replica had a similar pocket form in the first hour and also tore during the second hour of testing.
Acetic anhydride	All three replicas exhibited a large depression approximately 0.5 inches deep and 1.0 inches in diameter during the first hour. The CPE material became very elastic and would rebound after prodding with the blunt rod. Further changes were not observed during the test.
Acetone	All three replicas exhibited an indentation of approximately 0.4-0.5 inches. The material was not elastic as the indentation did not rebound to the original shape after prodding. Additionally, one swatch tore in the second hour.
bis(2-Chloroethyl) ether	All three replicas tore within one hour.
Carbon tetrachloride	The CPE swatches became elastic after exposure to CCl_4. A pocket indentation formed within the first hour that oscillated from a depression when prodded, to an elevated nipple when the rod was removed. These observations were made for all three replicas. No tearing or other physical changes were observed during the test.

(continued)

TABLE 8. DEGRADATION OF CHEMICAL-SOAKED CPE REPEATEDLY SUBJECTED TO PHYSICAL DEFORMATION (continued)

COMPOUND	COMMENTS/OBSERVATIONS
Ethylenediamine	One swatch tore lengthwise across the holder within one hour. The other swatches exhibited an indentation of 0.25 inches within the first hour that grew to 0.5 inches by the end of the test.
Isopropyl alcohol	All replicas exhibited an indentation that grew to a depth of 0.4-0.5 inches. Material rebounded (i.e., depth of pocket was reduced) when rod was withdrawn. No tearing or other physical changes were observed.
N-Nitrosodimethylamine	All replicas tore within the first hour.
Phenol	All swatches became very stiff and non-elastic within the first hour. All swatches tore during the final hour of testing.
O-Xylene	All replicas tore within one hour.

4.5 COMPARISON OF 20 MIL CPE TEST RESULTS AND POTENTIAL OPERATIONAL REQUIREMENTS FOR LSCPE USERS

In this section, the performance of 20 mil CPE as measured by the tests conducted in this project will be compared to a limited number of potential operational requirements that may confront the wearer of the LSCPE. The discussion in this section should not be taken as a firm recommendation for or against the use of 20 mil CPE in any specific situation. Determination of acceptability for specific situations should include the consideration by a qualified person of other factors-- e.g. temperature; probability of exposure; toxicity; type of exposure (immersion, splash, mist, vapor, etc.); surface area exposed; length of exposure; time available to escape, decontaminate, and doff the outer garment; other materials of construction of the garment; etc.-- in addition to those addressed in this report.

As described in the introduction, the reason for evaluating the performance of 20 mil CPE is that this polymer is the primary outer garment material of a prototype ensemble that is currently undergoing advanced development, mainly of the breathing apparatus, in an EPA/Army project. The ensemble has a self-contained breathing supply with a duration of 2.5 hours, so in a worst case the ensemble outer garment may be required to withstand 2.5 hours of chemical contact without degrading or permitting harmful permeation to occur. With this 2.5 hour service life in mind, the performance of CPE was examined in two areas:

One performance area was Permeation Breakthrough Resistance - Each CPE-chemical pair was placed in one of four categories based upon the breakthrough times observed in the permeation tests conducted in this project.

The second performance area was Tearing/Deformation Resistance - Each CPE-chemical pair was placed in one of two categories based upon the results obtained from the test in which CPE swatches were soaked in the test chemical and then repeatedly prodded with a blunt steel rod for up to two hours, as described in the procedures section.

The criteria for categorizing the test results and the actual categorization of results is shown in Table 9. As can be seen by examining Table 9, none of the ten compounds that were rated received category I (the best) ratings in both performance areas. It is possible that CPE's performance against carbon tetrachloride and isopropyl alcohol would, if additional permeation testing were done, receive a higher rating. Two tests by RMAL for carbon tetrachloride produced breakthrough times of <1 minute. However, there were also three breakthrough times generated by RMAL of approximately 200 minutes. These higher breakthrough times compared closely with those (mean breakthrough time = 207 min, n=2) obtained by Radian. In the case of isopropyl alcohol, RMAL obtained, in each of three tests, breakthrough times of <1 minute. Breakthrough times in excess of 480 minutes were obtained by Radian in each of three tests.

TABLE 9. CATEGORIZATION OF PERMEATION RESISTANCE AND TEARING/DEFORMATION RESISTANCE PERFORMANCE OF 20 MIL CPE

Chemical	Performance Area	
	(1) Tearing/Deformation Resistance	(2) Permeation Breakthrough# Resistance
1. Acetic acid	II	II
2. Acetic anhydride	II	II
3. Acetone	II	IV
4. Bis(2-chloroethyl)ether	III	II
5. Carbon tetrachloride	I	III*
6. Ethylenediamine	II	I
7. Isopropanol	I	III*
8. N-nitrosodimethylamine	III	II
9. Phenol	III	I
10.O-Xylene	III	II
	I No tearing of the three test swatches during 2 hours of prodding test.	I Mean breakthrough time > 2.5 hours.
	II One of three swatches tore during 2 hours of prodding test.	II Mean breakthrough times between 30 minutes and 2.5 hours.
	III Two or three test swatches tore within two hours of the prodding test	III Large variance in data (Isopropyl alcohol and carbon tetrachloride).
		IV Mean breakthrough times occurred before 30 minutes.

Rating based on laboratory mean breakthrough time with lowest relative standard deviation (RSD).
* Wide variation noted within and/or between laboratories.

Permeation resistance in excess of 2.5 hours, (150 minutes) was provided by 20 mil CPE against ethylenediamine, and phenol, but the resistance of CPE to deformation and tearing was degraded from contact with these chemicals.

For two compounds, acetic acid and acetic anhydride, the 20 mil CPE provided permeation breakthrough protection for more than one hour and two of three test swatches were not torn during the deformation tests.

Performance of CPE against o-xylene was marginal in terms of both permeation and degradation resistance.

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APPENDIX A

Designation: F 739 – 81

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Standard Test Method for RESISTANCE OF PROTECTIVE CLOTHING MATERIALS TO PERMEATION BY HAZARDOUS LIQUID CHEMICALS[1]

This standard is issued under the fixed designation F 739; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

Workers involved in the production, use, and tranportation of liquid chemicals can be exposed to numerous compounds capable of causing harm upon contact with the human body. The deleterious effects of these chemicals can range from acute trauma such as skin irritation and burn to chronic degenerative disease such as cancer. Since engineering controls may not eliminate all possible exposures, attention is often placed on reducing the potential for direct skin contact through the use of protective clothing that resists permeation, penetration, and degradation.

This method determines resistance to permeation only. Resistance to penetration and degradation should be determined by other methods.

1. Scope

1.1 This method covers the determination of the resistance of protective clothing materials to permeation by hazardous liquid chemicals under the condition of continuous liquid contact.

2. Applicable Documents

2.1 *ASTM Standards*:

E 105 Recommended Practice for Probability Sampling of Materials[2]

E 171 Specification for Standard Atmospheres for Conditioning and Testing Materials[2]

2.2 *Federal Standard*:

Fed. Std. No. 191, Method 5030.2 Measurement of the Thickness of Materials[3]

3. Summary of Method

3.1 The resistance of a protective clothing material to permeation by a hazardous liquid chemical is determined by measuring the breakthrough time then monitoring the subsequent permeation rate of the hazardous liquid through the clothing material.

3.2 In the permeation test apparatus, the clothing material acts as a barrier separating the hazardous liquid chemical from a collecting medium.

3.2.1 The collecting medium, which can be a liquid or a gas, is sampled and analyzed quantitatively to identify its concentration of hazardous chemical and thereby the amount of hazardous chemical that has permeated the barrier as a function of time after initial liquid contact.

3.2.2 By graphical representation or appropriate calculation, or both, both the breakthrough time and the permeation rate of the hazardous chemical are determined from the analytical data describing the amount of chemical that has permeated the barrier.

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[1] This method is under the jurisdiction of ASTM Committee F-23 on Protective Clothing, and is the direct responsibility of Subcommittee F 23.30 on Chemical Resistance.

Current edition approved Sept. 25, 1981. Published December 1981.

[2] *Annual Book of ASTM Standards*, Part 41.

[3] Available from Naval Publications and Forms Center, 5801 Tabor Ave., Philadelphia, Pa. 19120.

4. Significance and Use

4.1 This method is normally used to evaluate flat specimens from finished items of protective clothing and from materials that are candidates for items of protective clothing.

4.1.1 Finished items of protective clothing include gloves, arm shields, aprons, suits, hats, boots, respirators, and the like.

4.1.2 The phrase "specimens from finished items" encompasses seamed and other discontinuous regions as well as the usual continuous regions of protective clothing items.

4.2 By measuring breakthrough time, this method is used to estimate the duration of maximum protection provided by a protective clothing material under the condition of continuous contact.

4.3 By measuring the permeation rate, this method can be used to identify protective clothing materials that limit potential exposures to acceptable steady-state dermal contact levels.

NOTE 1—At the present time, such contact data are not available for most chemicals. They are yet to be developed by recognized occupational safety and health authorities by derivation from threshold limit values (TLVs), 50 % lethal doses (LD50), or other similar indexes.

4.4 The sensitivity of the test method in detecting low permeation rates is determined by the combination of: (*1*) the analytical technique selected, and (*2*) the ratio of material specimen area to collecting medium volume.

4.4.1 A sensitive analytical technique permits quantitative detection of the hazardous chemical in the collecting medium at the ppm to ppb level.

4.4.2 Higher ratios of material specimen area to collecting medium volume permit detection of earlier breakthrough times and lower permeation rates because higher concentrations of the hazardous chemical in the collection medium will develop in a given time period, relative to those which would occur at lower ratios.

5. Descriptions of Terms

5.1 *analytical technique*—a procedure whereby the concentration of a hazardous chemical in a collecting medium is quantitatively determined. Such procedures are often specific to individual chemical and collecting medium combinations. Applicable techniques can include UV and IR spectrophotometry, gas or liquid chromatography, colorimetry, and radioisotope tagging/scintillation counting.

5.2 *breakthrough time*—the elapsed time between initial contact of the hazardous liquid chemical with the outside surface of a protective clothing material and the time at which the chemical can be detected at the inside surface of the material by means of the chosen analytical technique.

5.3 *collecting medium*—a liquid or gas in which the hazardous liquid chemical is freely soluble to a saturation concentration greater than 0.5 weight % or volume %, and that does not affect the measured permeation rate.

5.4 *hazardous liquid chemical*—a liquid that can cause harm to the human body upon skin contact. The liquid can be either one component (that is, a neat liquid) or an aqueous or organic multicomponent solution.

5.5 *penetration*—the flow of a hazardous liquid chemical through zippers, stitched seams, and pinholes or other imperfections in a protective clothing material.

5.6 *permeation*—the process by which a hazardous liquid chemical moves through a protective clothing material on a molecular level. Permeation involves:

(*1*) sorption of molecules of the liquid into the contacted (outside) surface of a material;

(*2*) diffusion of the sorbed molecules in the material; and

(*3*) desorption of the molecules from the opposite (inside) surface of the material into the collecting medium.

5.7 *protective clothing material*—any material or combination of materials used in an item of clothing for the purpose of isolating parts of the body from direct contact with a hazardous liquid chemical.

5.8 *steady-state permeation*—the constant rate of permeation that occurs after breakthrough when all forces affecting permeation have reached equilibrium.

6. Apparatus

6.1 *Thickness Gage*, suitable for measuring thickness to the nearest 0.01 mm (or the nearest 0.001 in.), as specified in Fed. Std. No. 191 Method 5030.2, may be used to determine the thickness of each protective clothing material specimen tested.

6.2 *Analytical Balance*, accurate to ± 0.0005 g, used to determine weight per unit area of

each test specimen.

6.3 *Test Cell*—The test apparatus consists of a two-chambered cell for contacting the specimen with a hazardous liquid on the specimen's normal outside surface and with a collecting medium on the specimen's normal inside surface.

6.3.1 The test cell,[4] as shown in Fig. 1, is constructed of two sections of straight glass pipe, each nominally sized to a 51-mm (2.0-in.) diameter.[5] The section that is designated to contain the hazardous liquid chemical is 22 mm (0.85 in.) in length. The second section, which is designated to contain the collecting medium, is 35 mm (1.4 in.) or less in length as described in 6.4.1.5.

6.3.1.1 One end of each glass section is sealed closed (for example, with a glass disk equivalent in quality to that of the glass of the original sections).

6.3.1.2 The opposite end of each glass section retains the as-manufactured flared end.

6.3.1.3 Inlet and outlet ports with appropriate stopcock valves are added to each glass section as shown.

6.3.1.4 When assembled, the two glass sections are joined horizontally by aluminum or stainless steel flanges.

6.3.1.5 In tests where increased analytical sensitivity is required, a shorter length of glass section is used to contain the collecting medium. This reduces the contained volume and increases the ratio of material specimen area to the collecting medium volume.

6.3.2 A specimen is positioned between the two flanges as shown in Fig. 1. When the specimen is in place, the test cell is divided into two chambers.

6.3.3 Polytetrafluoroethylene (PTFE) gaskets are used in all joints.

7. Safety Precautions

7.1 Before carrying out this method, safety precautions recommended for handling each hazardous chemical of interest should be identified and reviewed to provide full protection to all personnel.

7.1.1 For carcinogenic, mutagenic, teratogenic, and other toxic (poisonous) chemicals, the work area should be isolated, well-ventilated, and meticulously clean. Involved personnel should be outfitted with protective clothing and equipment.

7.1.2 For corrosive or otherwise hazardous chemicals, involved personnel should, as a minimum, be outfitted with protective clothing and equipment.

7.2 Emergency equipment, such as a safety shower, eye wash, and self-contained breathing apparatus, should be readily accessible from the test area.

8. Test Specimen

8.1 Each protective clothing material specimen may consist of either a single layer or a composite of multiple layers that is representative of an actual protective clothing construction with all layers arranged in proper order. In each test, the specimen's normal outer surface shall contact the hazardous liquid chemical.

8.1.1 If, in a proposed design of an item of protective clothing, different materials or thicknesses of materials are specified at different locations, specimens from each location shall be tested.

8.1.2 If, in a proposed design, stitched-through or other type seams are specified, additional specimens containing such seams shall be tested.

8.2 Each material specimen to be tested shall have a minimum cross dimension of 60 mm (2.3 in.). A 75-mm diameter circle is convenient.

8.3 A minimum of three random specimens shall be tested for each material, composite, area (in the case of a heterogeneous design), or other condition. Random specimens shall be generated as described in Recommended Practice E 105.

9. Conditioning

9.1 Condition each protective clothing material specimen for a minimum of 24 h by exposure to a temperature of 21 ± 3°C (70 ± 5°F) and a relative humidity of 30 to 80 % as described in Specification E 171.

10. Procedure

10.1 Measure the thickness of each conditioned specimen to nearest 0.01 mm (or nearest 0.001 in.) and record.

10.2 Determine specimen weight per unit

[4] The test cell as shown is available from Pesca Lab Sales, P.O. Box 235, 226 Birch St., Kennett Square, Pa. 19348.

[5] Sections of borosilicate glass pipe, available from Corning Glass, Catalog No. 72-0702 (1-in. length), or equivalent, are satisfactory for this purpose.

area, plus or minus 5 %, and record.

10.3 Mount the first specimen in the test cell and assemble as shown in Fig. 1.

10.4 If the test is to be carried out at a nonambient temperature, immerse the assembled test cell in a constant-temperature chamber or a water bath at the test temperature.

10.5 Charge the collecting medium into the chamber to which the normal inside surface of the material specimen is exposed. Depending upon the combination of analytical technique and collecting medium selected, attach peripheral devices as appropriate (see Fig. 2).

10.5.1 If the test is to be carried out at a nonambient temperature and the collecting medium is a liquid, bring the medium to the test temperature before charging into the test cell.

10.5.2 The combination of analytical technique and collecting medium shall be selected to:

(*1*) maximize sensitivity of hazardous chemical detection, and

(*2*) represent actual occupational conditions as closely as possible.

10.5.2.1 Distilled water is preferred as a collecting medium when simulating perspiration on the inside surface of the material specimen. Consider alternative liquids only when the hazardous chemical is not fully soluble in distilled water as described in 5.3.

10.5.2.2 Nitrogen or helium is preferred as a collecting medium when simulating air on the inside surface of the material specimen. Consider alternative gases only when nitrogen or helium interferes with analytical detection of the hazardous chemical.

10.6 Stir or circulate the collecting medium continuously in the test cell so that a homogeneous mixture of permeated hazardous liquid is maintained.

10.7 Expeditiously charge the hazardous liquid chemical into the chamber of the test cell to which the normal outside surface of the material specimen is exposed. Fill the chamber to the fill mark on the stem. Begin timing the test when the addition of the hazardous chemical commences.

10.7.1 If the test is to be carried out at a nonambient temperature, bring the hazardous liquid chemical to the test temperature before being charged into the test cell.

10.8 Initiate sampling of the collecting medium, either continuously or discretely, and continue on a predetermined schedule. Promptly complete analysis of the sample for hazardous chemical content.

NOTE 2—The method chosen for collecting medium withdrawal shall be based on the technique selected for analytical detection. For example, UV spectroscopy is often used for continuous analysis of a sample stream (although compounding and curing agents often used in protective clothing materials can interfere) while gas chromatography requires the analyses of discrete samples.

When sampling using continuous-flow techniques, the flow of collecting medium should never be interrupted. This will minimize adsorption of permeated hazardous liquid on the cell walls.

10.9 Hold the volume of collecting medium in the test cell constant over the entire test.

10.9.1 In cases where samples are continuously withdrawn, analyzed and returned to the test cell, no provision for volume maintenance is necessary.

10.9.2 In cases where samples are withdrawn and discarded after analyses (primarily with a liquid medium), replenish the collecting medium volume with a volume of fresh medium equal to that of the withdrawn sample. Before its addition, bring the fresh medium to the temperature of the test cell.

NOTE 3—Replenishment of the collecting medium is necessary to simplify the calculation of hazardous chemical concentrations as described in 11.2.

10.10 Keep a record of the concentration of hazardous chemical found in the sample and the time which has elapsed between the initial liquid contact with the material specimen and withdrawal of the sample.

10.11 Discontinue sampling and terminate the test after a steady-state permeation rate has been established.

10.12 After the first material specimen has been tested, disassemble the test cell, thoroughly clean it, and prepare for another test.

10.13 Test additional material specimens until a minimum of three specimens per condition, as outlined in Section 8, have been tested.

11. Calculations

11.1 *Symbols*—The following symbols are used in the equations:

A = area of the material specimen contacted, m^2

c_{i-1}, c_i = concentrations of hazardous chemical

found in discrete samples number $i - 1$, i, mg/L

C_n = corrected concentration of hazardous chemical in the collecting medium at the time of discrete sample number n, mg/L

c_n = concentration of hazardous chemical found in discrete sample number n, mg/L

i = an indexing number assigned to each discrete sample taken, starting with $i = 1$ for the first sample

J = steady-state flux of permeating hazardous chemical, mg/m^2·s

s = slope of the curve depicting concentration of hazardous chemical versus elapsed time, mg/L·s

n = number of the most recent discrete sample taken

Q_s = rate of collecting medium make-up, mL/s

ΔT_i = time elapsed between the time of concentration c_{i-1} and concentration c_i, s

V_p = volume of collecting medium in the test cell prior to sampling, mL

V_s = volume of each discrete sample, mL

V_T = total volume of collecting medium in use, L

11.2 *Calculations for Systems Using Recycled Collecting Medium*—These calculations are applicable to a sampling methodology in which a portion of the collecting medium is removed from the cell, analyzed for permeant, and returned to the cell.

11.2.1 For each protective clothing material specimen tested, graph the concentration of hazardous chemical found in each sample of collecting medium as a function of the time elapsed between initial liquid contact with the specimen and withdrawal of the sample. Units shall be concentration in milligrams per litre versus time in seconds as shown in Fig. 3.

11.2.2 Calculate the steady-state flux of permeating hazardous chemical for each material specimen tested. This calculation shall be based on the value of the slope of the curve depicting hazardous chemical concentration versus elapsed time. The value of the slope of the curve shall be taken in the linear steady-state region denoting a constant rate of change as shown in Fig. 3, and the calculation is made using the equation: $J = SV_T/A$.

11.3 *Calculations for Systems Using Discrete Sampling and Replenishment with Fresh Collecting Medium*:

11.3.1 The concentrations of hazardous chemical found in samples of the collecting medium that has been previously sampled and then replenished with an equal volume of fresh collecting medium fluid are less than those representative of the total amount permeating due to the dilution effects. Therefore, a correction to account for these effects must be made before plotting the final test concentrations.

11.3.1.1 For each discrete sample, calculate a corrected concentration as follows:

$$C_n = c_n + \frac{V_s}{V_p} \sum_{i=1}^{n-1} c_i$$

Note that when $n = 1$, the value of the summation is zero.

11.3.2 Plot the corrected concentrations determined in this manner versus time, determine the slope, and calculate the permeation rate as described in 11.2.2.

11.4 *Calculations for Systems Involving Continuous Withdrawal of Collection Medium and Replenishment with Fresh Medium*:

11.4.1 Calculate a corrected concentration as follows:

$$C_n = c_n + \frac{Q_s}{V_p} \sum_{i=1}^{n-1} \left[\frac{c_{i-1} + c_i}{2} \right] \Delta T_i$$

11.4.2 Plot the corrected concentrations determined in this manner versus time, determine the slope, and calculate the permeation rate as described in 11.2.2.

12. Report

12.1 For each protective clothing material tested, prepare a report that describes the resistance of the material to the hazardous liquid chemical at test temperature.

12.1.1 Report the humidity of the laboratory atmosphere used to condition the samples.

12.1.2 Report the thickness of each material specimen to the nearest 0.01 mm (or nearest 0.001 in.). Also calculate and report the average thickness of the specimens tested for each material type.

12.1.3 Report weight per unit area of each specimen in grams per square metre to plus or minus 5 %. Also, calculate and report the average weight per unit area of the specimens tested for each material type.

12.1.4 Report the breakthrough time, in seconds, for each material specimen from the pre-

viously prepared graphical presentation of hazardous chemical concentration versus elapsed time. Also calculate and report an average breakthrough time of the specimens tested for each material type.

12.1.5 Report the steady-state flux, milligrams per square metre second of permeating hazardous chemical for each material specimen as calculated in 11.2.3. Also calculate and report the average steady-state flux for each material type.

12.1.6 Provide a copy of the graphical presentation prepared in accordance with 11.2.1 and used for deriving the breakthrough time and steady-state flux.

12.2 If a protective clothing material degrades rapidly after initial liquid contact such that no meaningful permeation data could be obtained, report this fact.

12.3 For each protecting clothing material tested, report both the collecting medium and the analytical technique (including its sensitivity) used.

13. Precision and Accuracy

13.1 A section on precision and accuracy will be prepared on accumulation of round-robin test data.

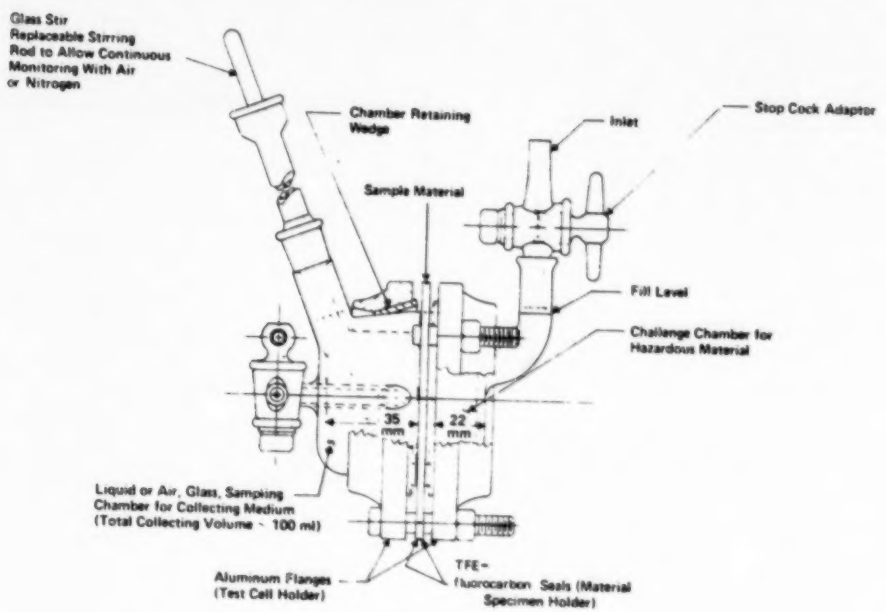

FIG. 1 Prototype ASTM Permeation Test Cell

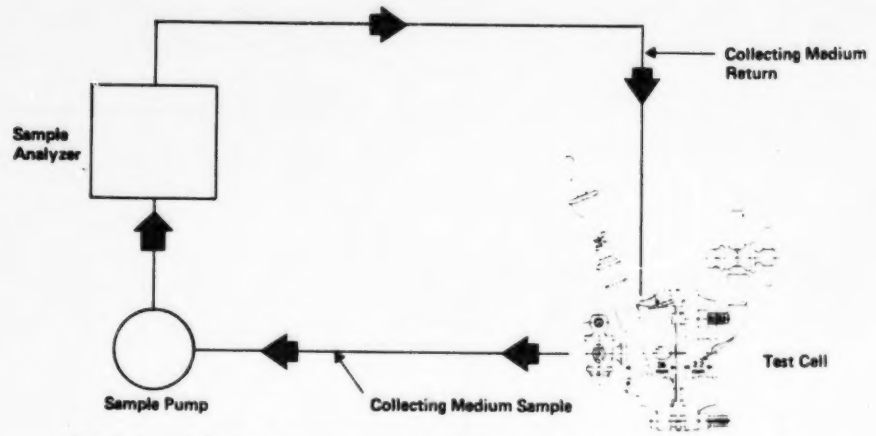

FIG. 2 Example Set-Up for Continuous Collecting Medium Sample Withdrawal, Analysis, and Return

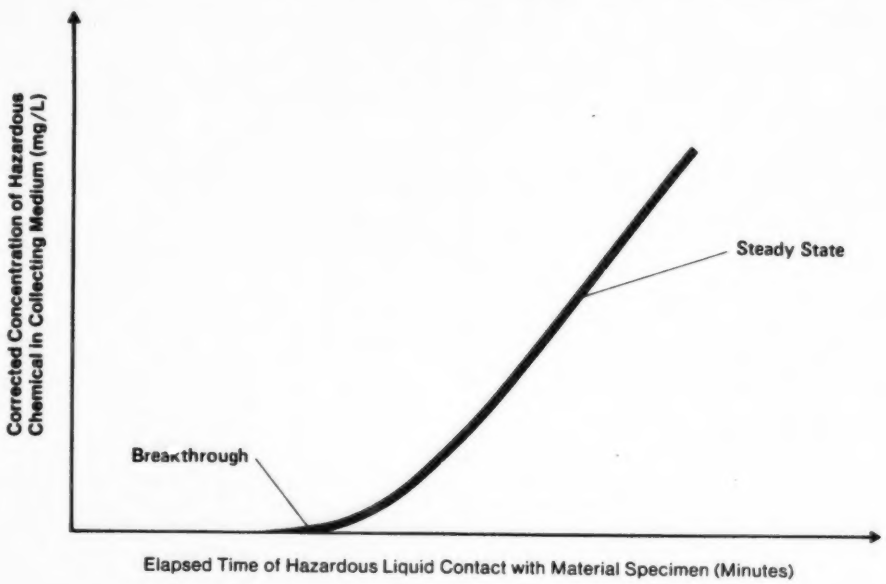
Corrected Concentration of Hazardous Chemical in Collecting Medium (mg/L)
Steady State
Breakthrough
Elapsed Time of Hazardous Liquid Contact with Material Specimen (Minutes)

FIG. 3 Typical Hazardous Liquid Permeation Through a Clothing Material Specimen

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APPENDIX B

SPECIFIC APPARATUS AND PROCEDURES USED FOR PERMEATION TESTS BY RMAL

A. Apparatus

Thickness gage, B.C. Ames #2 Comparater, B.C. Ames Co., Waltham, MA.
Balance, Mettler PE 160, Mettler Instrument Corp., Hightstown, N.J.

Test Cell, One-inch ASTM Permeation Test Cell, catalog #PTC 600, Pesce Lab Sales, Kennett Square PA.

Pump, closed loop stainless steel bellows pump, catalog #0715707, Foxboro Analytical, Foxboro, MA.

Tubing, 0.25 inch O.D. virgin Teflon tubing, Boulder Plastics, Boulder, CO.

Infrared spectrometer, Miran 1 General Purpose Gas Analyzer, Foxboro Analytical, Foxboro, MA.

Recorder, Fisher Recordall Series 5000 Strip Chart Recorder, Fisher Scientific Company, Pittsburgh, PA.

Syringes, various sizes, Hamilton Company, Reno, NV.

B. General Procedural Information

1. CPE swatch thickness - Swatch thickness was measured according to the procedure outlined in Federal Standard Number 191 Method 5030.2 (Determination of Thickness of Materials). All swatches measured 0.53 $\pm$ 0.01 mm.

2. CPE swatch weight - All swatches had a mass of 720 g/m^2 ($\pm$ 5%).

3. CPE sample conditioning - All swatches were conditioned at 21 $\pm$ 3^{o}C for at least 24 hours prior to testing.

4. Test temperature - All breakthrough and permeation tests were conducted at 21^{o}, except for ethylene oxide which was tested at 4^{o}C. The humidity was not recorded.

5. Analytical technique and detection limits - All determinations of breakthrough times and permeation rates were based on continuous

infrared spectroscopy analysis of the given test chemical in the collecting medium. Transmittance values were measured but were converted to absorbance values which are more directly related to concentration through Beers Law:

$$A_{IR} = eCl$$

where

A = absorbance = log of the fractional transmittance
e = slope of the calibration curve divided by the pathlength
C = concentration of the chemical in the collecting medium
l = spectral cell path length

The operating conditions for the Miran 1 infrared spectrometer are listed in Table B-1 along with the estimated detection limits.

6. Calibration and linearity of calibration curves - Calibration plots of absorbance versus standard concentrations were based on standards covering a minimum of three concentrations, but were often based on more. Calibration data for eight of the ten compounds tested are presented in Table B-1. An example of a calibration curve (for phenol) is presented in Figure 1.

TABLE B-1. MIRAN 1 INFRARED SPECTROMETER OPERATIONAL CONDITIONS

COMPOUND	WAVELENGTH (um)	CELL PATHLENGTH (m)	COLLECTION MEDIUM	ESTIMATED DETECTION LIMIT (ul/l)
Acetic acid	8.5	20.25	Nitrogen	5.6
Acetic anhydride	9.0	20.25	Nitrogen	2.1
Acetone	8.2	2.25	Nitrogen	1.1
bis(2-Chloroethyl) ether	8.8	20.25	Nitrogen	2.2
Carbon tetrachloride	12.6	20.25	Nitrogen	1.7
Ethylenediamine	13.0	22.25	Nitrogen	106
Isopropyl alcohol	8.7	8.25	Nitrogen	---
N-Nitrosodi-methylamine	8.7	20.25	Nitrogen	35
Phenol	8.5	20.25	Nitrogen	1.9
ortho-Xylene	9.6	20.25	Argon	29

TABLE B-2. RMAL CALIBRATION DATA FOR PERMEATION TESTS

COMPOUND	STANDARD INJECTED (ul)	DENSITY (mg/ul)	STANDARD CONCENTRATION (mg/l)	ABSORBANCE	SLOPE OF CALIBRATION CURVE	LINEAR REGRESSION CORRELATION COEFFICIENT
Acetic acid	0.5	1.049	0.092	0.10		
	0.5		0.092	0.089		
	1.0		0.184	0.18	0.554	0.993
	2.0		0.360	0.28		
	3.0		0.552	0.40		
	5.0		0.920	0.58		
	5.0			0.54		
Acetic anhydride	0.25	1.082	0.047	0.058		
	0.50		0.095	0.18		
	0.50		0.095	0.22	2.26	0.985
	1.0		0.190	0.39		
Acetone	5	0.790	0.692	0.12		
	10		1.39	0.21		
	10		1.39	0.24	0.162	0.997
	20		2.77	0.44		
	20		2.77	0.46		
	20		2.77	0.46		
bis(2-Chloroethyl) ether						

(continued)

TABLE B-2. RMAL CALIBRATION DATA FCR PERMEATION TESTS (continued)

COMPOUND	STANDARD INJECTED (ul)	DENSITY (mg/ul)	STANDARD CONCENTRATION (mg/l)	ABSORBANCE	SLOPE CALIBRATION CURVE	LINEAR REGRESSION CORRELATION COEFFICIENT
Carbon tetrachloride	0.2	1.594	0.056	0.14		
	0.5		0.140	0.23	0.738	0.875
	0.5		0.140	0.22		
	0.5		0.140	0.21		
	1.0		0.280	0.31		
Ethylene diamine	10	0.900	1.58	0.010		
	20		3.16	0.22		
	20		3.16	0.21	0.074	0.972
	25		3.95	0.28		
	30		4.74	0.31		
	50		7.89	0.51		
Isopropyl alcohol	0.2	0.786	0.078	0.015		
	0.5		0.069	0.036		
	0.5		0.069	0.060	1.11	0.996
	1.0		0.138	0.084		
	1.0		0.138	0.11		
	3.0		0.414	0.41		
	4.0		0.552	0.59		
N-Nitrosodimethyl-amine						

(continued)

TABLE B-2. RMAL CALIBRATION DATA FOR PERMEATION TESTS (continued)

COMPOUND	STANDARD INJECTED (ul)	DENSITY (mg/ul)	STANDARD CONCENTRATION (mg/l)	ABSORBANCE	SLOPE CALIBRATION CURVE	LINEAR REGRESSION CORRELATION COEFFICIENT
Phenol	0.5	1.072	0.094	0.051		
	1.0		0.188	0.14		
	1.0		0.188	0.11	0.337	0.996
	1.0		0.188	0.12		
	5.0		0.940	0.39		
	5.0		0.940	0.35		
	10		1.88	0.68		
Xylene	25	0.87	3.82	0.089		
	50		7.63	0.16		
	50		7.63	0.18	0.010	0.970
	100		15.3	0.26		
	100		15.3	0.26		
	200		30.5	0.37		

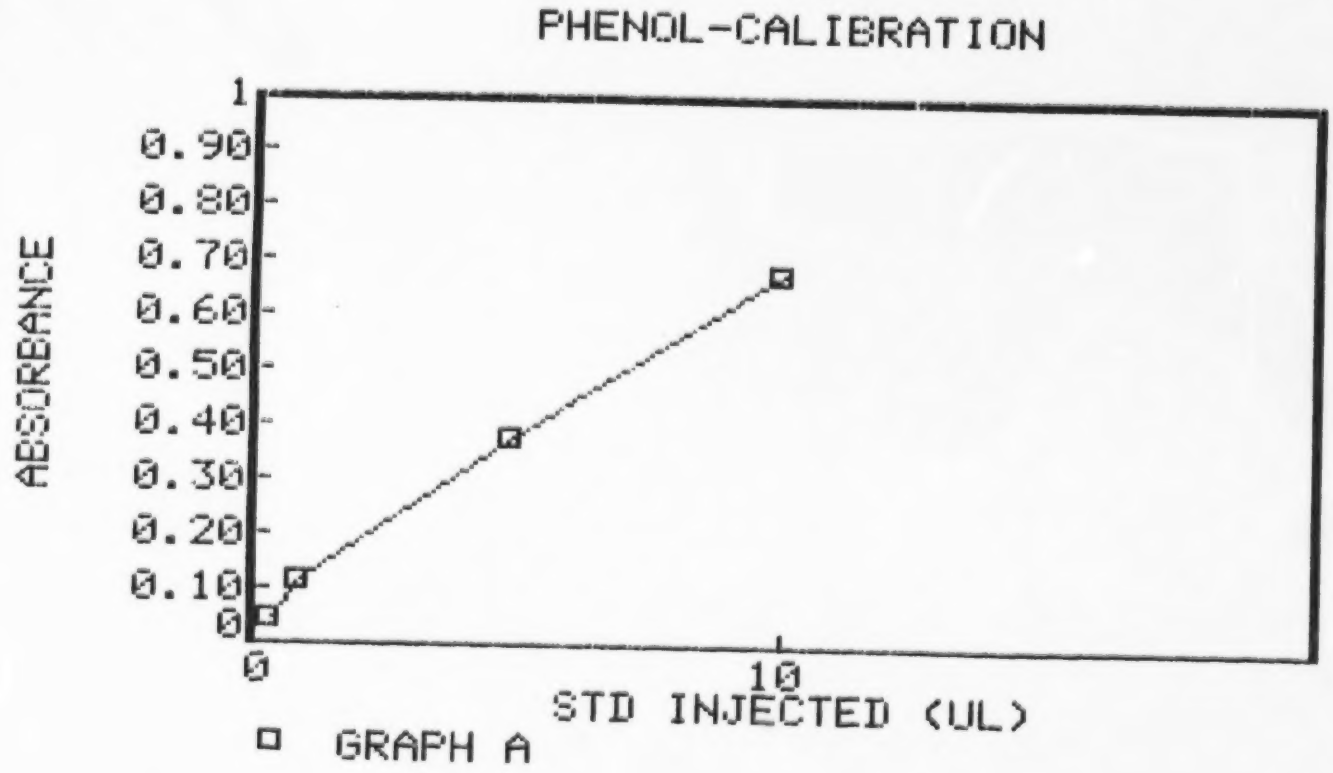

Figure B-1. Phenol Calibration.

C. RMAL Procedure

1. Cut the chlorinated polyethylene (CPE) swatches into circles with an approximate diameter of 1.5 inches (3.8 cm) as in Determination of Thickness of Textile Materials (Appendix D).

2. Record the ambient air temperature. If the temperature is not within the required limits (18-24°C), do not proceed with testing.

3. Mount the swatch in the permeation cell and secure with Teflon gaskets and flanges.

4. Insert the cell into the closed loop system diagramed below at the position indicated.

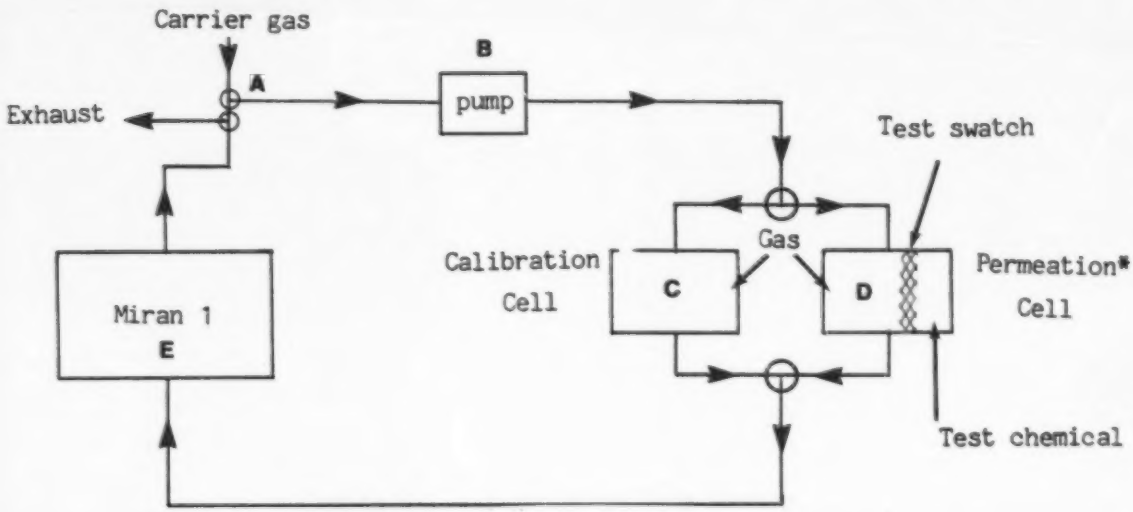

Figure B-2. Permeation Test System

*See Appendix A for a detailed drawing of the ASTM test cell.

5. Purge the system with carrier gas for 10 minutes by opening the appropriate valves. Make sure that the exhaust hood is on.

6. Close the system and allow carrier gas to circulate.

7. Obtain a scan of the entire IR spectrum monitored by the Miran 1 (2.5 to 14.5 um).

8. Inject an aliquot of the challenge chemical through the calibration port. Obtain a scan of the entire spectrum.

9. Compare the compound spectrum obtained in step 8 to the blank spectrum from step 7 and choose an appropriate maximum.

10. Set the Miran 1 to monitor the wavelength chosen in step 9 and purge the system with carrier gas until 100% transmittance is obtained. Adjust gains and offsets if required.

11. Standardize the system by sequentially injecting a minimum of three standards into the closed loop system. Record instrument response with the Fisher Recordall Strip Chart Recorder. Purge the system with carrier gas between each standard.

12. Record all instrument operational parameters on the strip chart paper.

13. Purge the system with carrier gas and charge the permeation cell with challenge chemical. Mark the strip chart recording when the cell is filled to the scribed line.

14. Monitor the IR response to detect breakthrough.

15. Continue to monitor permeation until the transmittance has dropped to 25%.

16. Purge the system and remove the challenge chemical and the CPE swatch from the permeation cell.

17. Verify instrument calibration by analyzing a calibration standard. The transmittance must agree with the original data point within 10%. If it does not, recalibration is required.

18. Verify the baseline by monitoring the instrument response while circulating carrier gas.

19. Complete steps #13 through #18 three times for each chemical type that is tested. Use fresh chemical and an unused swatch of CPE for each permeation test.

D. RMAL Calculations

Breakthrough time - Breakthrough is determined when three sequential data points all have an absorbance that is 0.01 units higher than the previous point. Obtain data points at least one minute apart if possible. For those compounds which have breakthrough times less than 15 minutes, apply the above test to data points collected at 30 second intervals.

APPENDIX C

AMERICAN NATIONAL STANDARD

ASTM D 471 – 79

AMERICAN SOCIETY FOR TESTING AND MATERIALS
1916 Race St., Philadelphia, Pa. 19103
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Standard Test Method for RUBBER PROPERTY—EFFECT OF LIQUIDS[1]

This standard is issued under the fixed designation D 471; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

1. Scope

1.1 This method measures the comparative ability of rubber and rubber-like compositions to withstand the effect of liquids. It is designed for testing: specimens of elastomeric vulcanizates cut from standard sheets (see Recommended Practice D 3182); specimens cut from fabric coated with elastomeric vulcanizates (see Method D 751); or finished articles of commerce (see Recommended Practice D 3183). The method is not applicable to the testing of cellular rubbers, porous compositions, and compressed asbestos sheet except as provided in Note 5.

1.2 In view of the wide variations often present in service conditions, this accelerated test may not give any direct correlation with service performance. However, the method yields comparative data on which to base judgment as to expected service quality and is especially useful in research and development work.

2. Applicable Documents

2.1 *ASTM Standards:*

D 412 Tests for Rubber Properties in Tension[2]

D 751 Testing Coated Fabrics[3]

D 1193 Specification for Reagent Water[4]

D 1415 Test for Rubber Property—International Hardness[5]

D 2240 Test for Rubber Property—Durometer Hardness[6]

D 3182 Recommended Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets[5]

D 3183 Recommended Practice for Rubber—Preparation of Pieces for Test from Other Than Standard Vulcanized Sheets[5]

3. Description of Test

3.1 This method provides a procedure for exposing test specimens to the influence of liquids under definite conditions of temperature and time. The resulting deterioration is determined by noting the changes in physical properties before and after immersion in the test liquid.

4. Test Conditions

4.1 *Temperature*—Unless otherwise specified the test temperature shall be one of the following, depending upon the anticipated service temperature:

Temperature, °C
−75 ± 2
−55 ± 2
−40 ± 2
−25 ± 2
−10 ± 2
0 ± 2
23 ± 2
50 ± 2
70 ± 2
85 ± 2

[1] This method is under the jurisdiction of ASTM Committee D-11 on Rubber and is the direct responsibility of Subcommittee D11.10 on Physical Testing.

Current edition approved March 30, 1979. Published May 1979. Originally published as D 471 – 37 T. Last previous edition D 471 – 78.

[2] *Annual Book of ASTM Standards*, Parts 35, 37, and 38.

[3] *Annual Book of ASTM Standards*, Part 38.

[4] *Annual Book of ASTM Standards*, Parts 20, 21, 22, 26, 29, 31, 37, 40, and 43.

[5] *Annual Book of ASTM Standards*, Part 37.

[6] *Annual Book of ASTM Standards*, Parts 35 and 37.

Temperature, °C
100 ± 2
125 ± 2
150 ± 2
175 ± 2
200 ± 2
225 ± 2
250 ± 2

4.1.1 When the temperature of the testing room is other than the standard 23 ± 2°C the temperature of test shall be reported.

4.2 *Immersion Periods*—The following immersion periods are recommended:

22 h
46 h
70 h
166 h
670 h

The choice of the immersion period will depend upon the nature of the vulcanizate, the test temperature, and the liquid to be used. To obtain information on the rate of deterioration it is necessary to make determinations after several immersion periods. The tolerance for any immersion period shall be ±15 min or ±1 % of the immersion period, whichever is greater.

4.3 *Light*—Immersion tests shall be made in the absence of direct light.

5. Standard Test Liquids

5.1 For purpose of test, it is usually desirable to use the liquid with which the vulcanizate will come in contact in service. For comparative tests with liquids of unknown or doubtful composition, samples of liquid from the same drum or shipment shall be used. Many commercial products, particularly those of petroleum origin, are subject to sufficient variation that it is not practical to use them for test liquids. It is then advisable to use a standard test liquid, such as described in 5.1.1 and 5.1.2, covering the range of properties that may be encountered in the particular service.

5.1.1 *ASTM Oils*—The test shall be conducted in one of the petroleum-base ASTM oils (Note 1) specified in Table 1 which has its aniline point nearest that of the oil with which the vulcanizate is expected to come in contact in service except as indicated in 5.1.3.

NOTE 1—The aniline point of a petroleum oil appears to characterize the swelling action of that oil on synthetic rubber. In general, the lower the aniline point, the more severe the swelling action by the oil. The oils specified in Table 1 cover a range of aniline points commonly found in lubricating oils.

5.1.2 *ASTM Reference Fuels*—When gasolines are to be encountered in service, the test shall be conducted in one of the ASTM Reference Fuels (Note 2) specified in Table 2, except as indicated in 5.1.3.

NOTE 2—The ASTM Reference Fuels in Table 2 have been selected to provide the maximum and minimum swelling effects produced by commercial gasolines. Reference Fuel A has a mild action on elastomeric vulcanizates and produces results of the same order as low-swelling gasolines of the highly paraffinic, straight-run type. Reference Fuel B has a more severe swelling action on elastomeric vulcanizates and exceeds the swelling action of commercial gasoline. Reference Fuel C has a more severe swelling action which is typical of these experienced with highly aromatic premium grades of automotive gasolines.

5.1.3 *Service Fluids*—Some commercial oils and fuels (Table 3) are either non-petroleum or are compounded from special petroleum hydrocarbon fractions or mixtures of petroleum hydrocarbon fractions and other ingredients resulting in materials having properties beyond the range of the reference fluids listed in Tables 1 and 2. Immersion tests of elastomeric vulcanizates that are to come into contact with such oils and fuels should be made in the actual service fluid.

5.1.4 *Type IV Reagent Water*—Since the purity of water varies throughout the world, all water immersion tests are to be conducted in Type IV Reagent Water in accordance with Specification D 1193.

6. Preparation of Sample

6.1 Except as otherwise specified in the applicable specifications, specimens shall be prepared in accordance with the requirements of Recommended Practices D 3182 and D 3183.

7. Apparatus

7.1 *For Nonvolatile Liquids*—A glass test tube having an outside diameter of 38 mm and an over-all length of 300 mm fitted loosely with a stopper (Note 3) shall be used.[7] Glass beads shall be used in the liquid as a bumper and to separate the specimens.

7.2 *For Volatile Liquids*—The test tube described in 7.1 shall be tightly fitted with a

[7] Test tubes 9205-G77 (package of 12) and 9205-G78 (case of 48), available from A. H. Thomas Co., Vine Street at Third, P.O. Box 779, Philadelphia, Pa. 19105, and from Precision Scientific, 3737 West Cortland St., Chicago, Ill. 60647 as part No. LG 226.

stopper (Note 3) and a reflux condenser. An air-cooled reflux condenser shall be used for immersion tests with ASTM Oils No. 1,[8,9] 2, 3, and 5[9] at test temperatures of 125°C or more. This condenser shall be a glass tube, approximately 750 mm in length and 15 mm in outside diameter, tightly fitted to the immersion tube by means of a stopper (Note 3). The bottom of the condenser tube shall project about 12 mm below the stopper. A suitable reflux condenser will be used for Type IV Reagent Water immersion tests. Glass beads shall be used as in 7.1.

NOTE 3—The stopper shall not contaminate the test liquid. When in doubt, cover the stopper with aluminum foil.

8. Test Specimens—Change in Mass or Volume

8.1 The standard specimen shall be rectangular having dimensions of 25 by 50 by 2.0 ± 0.1 mm (1 by 2 by 0.08 ± 0.005 in.). Specimens from commercial articles shall be the thickness of the material as received when they are less than 2 mm (0.08 in.); otherwise, they should be buffed to a thickness of 2.0 ± 0.1 mm (0.08 in.). Data obtained on test specimens having different original thicknesses are not comparable.

9. Procedure for Change in Mass

9.1 Three specimens of a single composition shall be tested. Weigh each test specimen in air to the nearest 1 mg, M_1, and immerse them in the test tube (see 7.1) containing 100 cm^3 of the test liquid (Note 4). Each test specimen is to be separated from any adjacent test specimen and the walls of the test tube by approximately 6 mm; for example, by perforated glass beads[10] as shown in Fig. 2. The materials used to suspend and separate the specimens shall not affect the test liquid or the rubber.

NOTE 4—Test liquids must not be reused.

9.1.1 For nonvolatile liquids, the test tube assembly shall be conditioned at the specified temperature within the limits given in 4.1 for the specified length of time. A check of the actual temperature of the test liquid contained in the test tube should be made to assure that the liquid is within the test temperature specified.

9.1.2 For volatile liquids, the test tube assembly shall be fitted with a reflux condenser (see 7.2) and conditioned at the specified temperature within the limits given in 4.1 for the specified length of time. A check of the actual temperature of the test liquid contained in the test tube should be made to assure that the liquid is within the test temperature specified.

9.2 After the immersion test has proceeded for the required length of time, remove the test specimens. If the immersion has been at an elevated temperature, cool the test specimens to room temperature by transferring them to a cool clean portion of the test liquid for 30 to 60 min. Then dip the specimens quickly into acetone, blot lightly with filter paper free of lint and foreign material, place them immediately in a tared, stoppered weighing bottle, and determine the mass after test, M_2, of each specimen. After weighing, again immerse the test specimen in the test liquid if data are desired on the progressive changes which occur with increasing time of immersion. It is important that each manipulation take place promptly with the least possible lapse of time, allowing no more than 30 s for each step, except the actual weighing in the tared, stoppered weighing bottle.

10. Procedure for Change in Volume

10.1 *Water Displacement Method for Water Insoluble Liquids and Mixed Liquids*—Three specimens shall be tested. Weigh each test specimen (Note 5) in air, M_1, to the nearest 1 mg (Note 5) and then weigh each specimen immersed in distilled water, M_3, at room temperature. Quickly dip each specimen in alcohol, to remove water blot dry with filter paper free of lint and foreign material, and place in the immersion apparatus described in 7.1 or 7.2. Add 100 cm^3 of liquid (Note 4) to the test tube

[8] Information about ASTM Oil No. 1 is included in Research Report RR: D-11-1004 and is available on loan from ASTM Headquarters, 1916 Race St., Philadelphia, Pa. 19103.

[9] ASTM Oil No. 1 may be ordered from MZF Asso., 11200 Homedale St., Los Angeles, Calif. 90049.

ASTM Oils No. 2, 3, and 5 are distributed domestically for Sun Oil Co. by R. E. Carroll, P.O. Box 139, Trenton, N.J. 08601. Overseas requests should be directed to Sun Trading and Marine Transporting Co., 1845 Walnut St., Philadelphia, Pa. 19103.

[10] Hollow 6-mm perforated glass beads may be purchased from Fisher Scientific Co., 590 Lincoln St., Waltham, Mass. 02154. These are identified as Catalog Item No. 11-311C.

ness as *L*, *W*, and *T*, respectively. If the immersion liquid tends to volatilize at room temperature, the measurements should be completed within 1 min after removing the test specimens from the liquid at room temperature.

12. Procedure for Change in Mass with Liquid on One Surface Only

12.1 *Scope*—This method provides a procedure for exposing thin sheet materials (rubber-coated fabrics, diaphragms, etc.) that are exposed to the immersion liquid on only one suface under definite conditions of time and temperature.

12.2 *Test Specimen*—A disk 60 mm (2.4 in.) in diameter and thickness of thin sheet or rubber coated fabric being tested.

12.3 *Apparatus*—The essential features of the apparatus are illustrated in Fig. 1 and consist of a base plate, *A*, an open-ended cylindrical chamber, *B*, which is held tightly against the test specimen, *C*, by wing nuts, *D*, mounted on bolts, *E*. During the test, the opening in the top of the chamber is tightly closed by a suitable plug, *F*.

12.4 *Procedure*—Weigh the test specimen in air to the nearest 1 mg, M_1, and place in the apparatus as indicated in Fig. 1. Fill the chamber of the apparatus with the test liquid to a depth of 15 mm and complete the test at the specified conditions as described in 4.1 and 4.2. At the end of the required immersion period, bring the apparatus to Standard Room Temperature, remove the test liquid and release the test specimen. Remove any excess liquid from the surface of the specimen by wiping or blotting with filter paper free of lint and foreign material. Determine the mass of the specimen to the nearest 1 mg and record as M_2. If the immersion liquid tends to volatilize at room temperature, complete the measurement within 2 min after removal of the test specimen from the liquid.

13. Procedure for Determining Mass of Soluble Matter Extracted by the Liquid

13.1 This method is applicable for immersions primarily in volatile-type immersion liquids such as the ASTM Reference Fuels (see 5.1.2). The immersions are conducted in accordance with Section 9, including 9.1, 9.1.1, and 9.1.2.

13.2 At the end of the required immersion period, remove the test specimens from the test tube and dry to a constant mass at a temperature of approximately 40°C and an absolute pressure below 20 kPa (150 mmHg). Express the change in mass as a percentage of the original mass of the test specimen.

13.3 Transfer the liquid in which the test specimens have been immersed to a suitable vessel and wash the test specimen with 25 cm^3 of fresh liquid which is transferred to the same vessel. Evaporate the liquid and dry the residue to constant mass in a vacuum and weigh.

14. Changes in Tensile Strength, Elongation, and Hardness

14.1 *Original Properties*—The original tensile strength, ultimate elongation, and hardness shall be determined, using duplicate specimens cut from the sheet or article adjacent to those which are to be immersed in the liquid.

14.2 *Immediate Deteriorated Properties*—For determining the tensile strength, ultimate elongation, and hardness, prepare three test specimens from flat vulcanized sheets 2.0 ± 0.1 mm (0.08 ± 0.005 in.) (see 8.1) using Die C of Method D 412. Measure the thickness of each test specimen, place the specimens in the test tube described in 7.1, and add 150 cm^3 of liquid (Note 4) to the test tube. Complete the immersion test in accordance with 9.1.1 or 9.1.2. At the end of the required immersion period, remove the specimens from the test tubes, and if necessary, cool immediately to room temperature in a fresh sample of the same liquid for 30 to 60 min. Then quickly dip each specimen in acetone and blot lightly with filter paper free of lint and foreign material. Immediately apply bench marks and determine the tensile strength and ultimate elongation in accordance with Method D 412, using the original unimmersed thickness or cross-sectional area. Determine the hardness of each specimen in accordance with Method D 1415 or Method D 2240. The time interval between removal from the cold liquid and testing shall not be less than 2 nor more than 3 min.

14.3 *Properties After Evaporation of Test Liquid*—For determining the tensile strength and ultimate elongation of specimens after evaporation of the immersion liquid, immerse the specimens in the test liquid in accordance with

and complete the immersion test as described in 9.1.1 or 9.1.2. At the end of the required immersion period, remove each specimen from the test tube. Cool the specimens to room temperature by transferring them to a cool clean portion of the test liquid for 30 to 60 min; then dip quickly in acetone at room temperature, blot lightly with filter paper, place in a tared, stoppered weighing bottle, and weigh, M_3 (Note 9). Remove each specimen from the bottle and weigh in distilled water, M_4, in immediate consecutive procedure to determine the water displacement after immersion (Notes 5, 6, and 7). It is important that each manipulation following removal of the specimen from the test medium take place promptly with the least possible lapse of time. When utilizing liquids that tend to volatilize at room temperature, no more than 30 s should elapse between removal from the test liquid and stoppering the weighing bottle, and no more than an additional 30 s between removal from the weighing bottle and immersion in water.

NOTE 5—It is important that all air bubbles clinging to the test specimen be removed before weighing in distilled water. If, in the course of weighing, air bubbles appear on the surface of the specimen, or the computed volume changes 0.5 % in 5 min, the specimen is too porous to permit volume determination in this manner. In that case, the initial volume of the specimen, if the latter is a simple geometrical solid, can be determined from the over-all dimensions by employing an appropriate mensuration formula, and the same procedure followed in determining the volume after the immersion test. Or, if volume increase occurs principally in the thickness dimension, a simple change in thickness may be substituted for change in volume. If in the course of weighing the test specimen floats, AISI No. 316 stainless steel can be used as ballast to immerse the test specimen in the test liquid. In those cases where ballast is needed to submerge the specimen, the following procedure may be used:

(*1*) Weigh the specimen with ballast in water.

(*2*) Weigh the ballast alone in water.

(*3*) Determine the difference between the weighings and proceed with the calculations.

NOTE 6—A jolly spring balance, adequately shielded from air currents, may be used for making these determinations. When utilizing liquids that tend to volatilize at room temperature, no more than 30 s should elapse between removal from the test liquid and completion of the weighing operation.

NOTE 7—When data are desired on the progressive changes occurring with increasing time of immersion, the test specimen shall be immediately removed from the water after making this determination, dipped quickly into acetone at room temperature, blotted lightly with filter paper and replaced in the test liquid with the least possible lapse of time.

NOTE 8—The alcohol dip prior to immersion and the acetone dip after immersion shall be omitted when Type IV Reagent Water is employed as the test medium.

NOTE 9—When the viscosity of the immersant is such that the oil cannot be cleaned off the specimen by blotting, a more vigorous wiping procedure should be employed.

NOTE 10—This procedure may not be suitable for immersion liquids (other than water) which are readily miscible with water or react with it. For such liquids that are not too viscous or volatile at room temperature, weighings M_2 and M_4 may be made in the immersion liquid instead of water with weighing M_4 made in a fresh portion of the immersion liquid. These values are then used in calculating volume change using the formula described in 15.2.

If this is not practicable, the same procedure is used as for the water displacement method except that the final weighing M_4 is omitted and change in volume is calculated from the following formula where d is the density of the immersion liquid at the Standard Laboratory Room Temperature:

$$\text{Change in volume, percent} = [(M_3 - M_1)/d(M_1 - M_2)] \times 100$$

This formula may be only approximate if the immersion liquid is a mixture, because the density of the absorbed liquid may differ from that of the bulk. Also the density of any matter extracted from the rubber may differ from that of the immersion liquid.

11. Dimensional-Change Method for Water-Insoluble Liquids and Mixed Liquids

11.1 *Dimensional Change Method*—Measure the original length and width of the three test specimens used in 10.1 to the nearest 0.5 mm using an average of three measurements in each direction recording as L_0 and W_0, respectively. Measure the thickness of each test piece using a dial micrometer as described in 3.4 (Note 2) Method D 412, as T_0. Quickly dip each specimen in alcohol, to remove water, blot dry with filter paper free of lint and foreign material, and place in the immersion apparatus described in 7.1 and 7.2. Add 100 cm^3 of liquid (Note 4) to test tube and complete the immersion test as described in 9.1.1 or 9.1.2. At the end of the required immersion period, remove each specimen from the test tube and bring to room temperature by transferring them to a cool clean portion of the test liquid for 30 to 60 min. Dip the specimens quickly in acetone at room temperature, blot lightly with filter paper, and promptly remeasure as described above, recording the immersed length, width, and thick-

14.2. At the end of the required immersion time, remove the specimens and if necessary cool them to room temperature in a fresh sample of the same liquid for 30 to 60 min. Then suspend the specimens for 4 ± ¼ h in air at room temperature shielded from drafts. For specimens that have been immersed in a non-volatile liquid, dip them quickly in acetone, and blot lightly with filter paper. Immediately apply bench marks and determine tensile strength and ultimate elongation in accordance with Method 412 using the original thickness or cross-sectional area, or both of the untreated specimens.

15. Changes in Breaking Strength, Burst Strength, Tear Strength, and Adhesion for Coated Fabrics

15.1 *Original Properties*—The original properties for breaking strength, burst strength, tear strength, and adhesion shall be determined in accordance with Methods D 751, using duplicate specimens cut from the coated fabric adjacent to those which are to be immersed in the liquid.

15.2 *Immediate Deteriorated Properties*—For determining breaking strength, burst strength, tear strength, and adhesion, prepare the number of specimens specified for each parameter in Methods D 751. Place the specimens in the test tube described in 6.1 and add 150 cm^3 of liquid (Note 4) to the test tube. Complete the immersion test in accordance with 8.1.1 or 8.1.2. At the end of the required immersion period, remove the specimens from the test tubes, and if necessary, cool immediately to room temperature in a fresh sample of the same liquid for 30 to 60 min. Then quickly dip each specimen in acetone and blot lightly with filter paper free of lint or foreign material. Measure the required physical properties in accordance with Methods D 751. The time interval between removal from the cold liquid and testing shall not be less than 2 min nor more than 3 min.

15.3 *Properties After Evaporation of Volatile Test Liquid*—For determining the breaking strength, burst strength, tear strength, and adhesion of specimens after evaporation of a volatile immersion liquid, immerse the specimens in the test liquid in accordance with 14.2. At the end of the required immersion time, remove the specimens and suspend them in a circulating-air oven at a temperature of 70 ± 2°C for a period of 2 h. At the end of the drying period, remove the specimens from the oven and allow them to cool to room temperature. The time interval between removal from the oven and testing shall not be less than 60 min nor more than 120 min.

16. Calculations

16.1 Calculate the change in mass (see Section 9) as follows:

$$\text{Change in mass, \%} = [(M_2 - M_1)/M_1] \times 100$$

where:

M_1 = initial mass of specimen, g, and
M_2 = mass of specimen, g, after immersion.

16.2 Calculate the change in volume (see Section 10) as follows:

$$\text{Change in volume, \%} = \frac{(M_3 - M_4) - (M_1 - M_2)}{M_1 - M_2} \times 100$$

where:

M_1 = initial mass of specimen in air, g,
M_2 = initial mass of specimen in water, g
M_3 = mass of specimen in air after immersion, g, and
M_4 = mass of specimen in water after immersion, g.

16.3 Calculate the change in dimensions (see Section 11) as follows:

$$\text{Change in length, \%} = \Delta L = [(L - L_o)/L_o] \times 100$$

where:

L_o = initial length of specimen, and
L = length after immersion.

Calculate change in width, ΔW, or thickness, ΔT, in a similar manner.

16.4 Calculate change in mass (see Section 12) as follows:

$$\text{Change in mass per unit surface, kg/m}^2 = 1000\,(M_2 - M_1)/A$$

where:

M_1 = initial mass of test specimen, g,
M_2 = final mass of test specimen, g, and
A = area of the test specimen in actual contact with the liquid, mm^2.

16.5 Calculate tensile strength of the specimens both on immediate deteriorated properties (see 14.2) and properties after evaporation

of test liquid (see 14.3) as described in Method D 412.

$$\text{Tensile stress} = F/A$$

where:

F = observed force, and

A = original unstretched cross-sectional area of the test specimen before immersion.

16.6 Calculate breaking strength, burst strength, tear strength, and adhesion both on the immediate deteriorated properties (see 15.2) and properties after evaporation of volatile test liquid (see 15.3) as described in Methods D 751.

17. Report

17.1 State that the test was conducted in accordance with ASTM Method D 471 and report the following:

17.1.1 Date and temperature of testing room (see 4.1),

17.1.2 Duration, temperature, and date of vulcanization of the test specimens,

17.1.3 Dates of the various periods of exposures,

17.1.4 Immersion liquid used,

17.1.5 Temperature of exposure,

17.1.6 Exposure period.

17.1.7 All observed and recorded data, to include the type of properties being reported,

17.1.8 Results calculated in accordance with Section 16, and

17.1.9 Statement of condition of exposed specimens from visual and manual examination.

17.1.10 Report which test method was used for determination of hardness.

TABLE 1 ASTM Reference Oils[A]

	Oil No. 1	Oil No. 2	Oil No. 3	Oil No. 5	ASTM Methods[A]
Aniline point, °C	124.0 ± 1.0	93.0 ± 3.0	70.0 ± 1.0	115.0 ± 1.0	D 611
Kinematic viscosity, mm^2/s (cSt)	18.7 to 21.00[B]	19.2 to 21.5[B]	31.9 to 34.1[C]	10.78 to 11.88[B]	D 445
Flash point min, °C	243	240	163	243.0	D 92

[A] These designations refer to the following methods of the American Society for Testing and Materials:
D 92, Test for Flash and Fire Points by Cleveland Open Cup[11]
D 611, Test for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents[11]
D 445, Test for Viscosity of Transparent and Opaque Liquids (Kinematic and Dynamic Viscosities)[11]

[B] Measured at 99°C.

[C] Measured at 37.8°C.

TABLE 2 ASTM Reference Fuels[12]

Reference Fuel A	*Iso*octane[A]
Reference Fuel B	*Iso*octane,[A] 70 volume % Toluene,[B] 30 volume %
Reference Fuel C	*Iso*octane,[A] 50 volume % Toluene,[B] 50 volume %
Reference Fuel D	*Iso*octane,[A] 60 volume % Toluene,[B] 40 volume %

[A] *Iso*octane conforming to Annex A2.8, Motor Fuels Section, of the *Annual Book of ASTM Standards*, Part 47.

[B] Toluene conforming to ASTM Specification D 362, for Industrial Grade Toluene.[13]

[11] *Annual Book of ASTM Standards*, Part 40.

[12] Reference Fuels A, B, C, and D are available from Phillips Petroleum Co., Customer Service Center, Drawer "O", Borger, Tex. 79007.

[13] *Annual Book of ASTM Standards*, Part 29.

TABLE 3 ASTM Service Fluids

Composition	Mass, %
Service Fluid No. 101[A]	
di-2-ethyl hexyl sebacate	99.5
phenothiazine	0.5
Service Fluid No. 102[14]	
ASTM Oil No. 1	95
hydrocarbon compound oil additive containing 29.5 to 33.5 mass % sulfur, 1.5 to 2.0 mass % phosphorus, and 0.7 mass % nitrogen[B]	5

[A] This test fluid is available as Anderol 774 from Tenneco Chemicals, Inc., Intermediates Div., P.O. Box 2, Turner Place, Piscataway, N.J. 08854. Physical and chemical requirements are as follows:

[B] Hydrocarbon compound oil additive having a kinematic viscosity 9.18 to 10.22 mm^2/s (9.18 to 10.22 cSt at 98.9°C), a Cleveland Open Cup Flash Point of 45°C min, and a specific gravity at 16°C of 1.065 ± 0.015.

Test	Value	ASTM Methods[C]
Kinematic viscosity, mm^2/s (cSt)		
At 37.8°C	12.7 ± 0.2	D 445
At 99°C	3.3 ± 0.1	
Flash Point, min, Cleveland Open Cup, °C	215	D 92
Specific gravity at 25°C	0.9130 ± 0.003	D 1217
Acid number, maximum increase from original after oxidation for 72 h at 176°C	2	D 1980

[C] These designations refer to the following methods of the American Society for Testing and Materials:
D 92, Test for Flash and Fire Points by Cleveland Open Cup[11]
D 445, Test for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)[11]
D 1217, Test for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer[15]
D 1980, Test for Acid Value of Fatty Acids and Polymerized Fatty Acids[13]

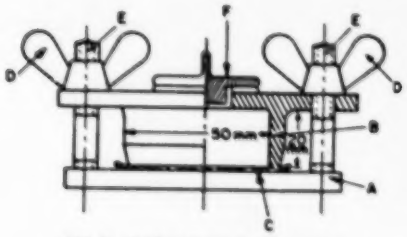

FIG. 1 Surface Immersion Apparatus.

[14] This hydrocarbon oil additive is available from Lubrizol Corp., 29400 Lakeland Blvd., Wickliffe, Ohio 44092 and is known as Anglamol 99.

[15] *Annual Book of ASTM Standards*, Part 23.

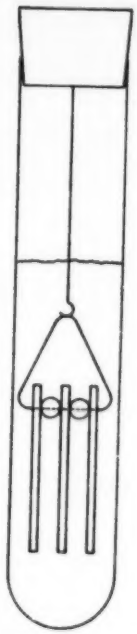

FIG. 2 Method of Separation.

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